PREPARATION AND CHARACTERIZATION OF WASTE SILK FIBER REINFORCED POLYMER COMPOSITES

A THESIS SUBMITTED IN PARTIAL FULFILMENT

OF THE REQUIREMENTS FOR THE DEGREE OF

Master of Technology (Research)

In

Mechanical Engineering

By

SANYUKTA GUPTA

Department of Mechanical Engineering

National Institute of Technology

Rourkela - 769008

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DEPARTMENT OF MECHANICAL ENGINEERING
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CERTIFICATE

This is to certify that the thesis entitled "**PREPARATION AND CHARACTERIZATION OF WASTE SILK FIBER REINFORCED POLYMER COMPOSITES**", submitted by **Ms. SANYUKTA GUPTA** in partial fulfillment of the requirements for the award of **Master of Technology (Research) Degree in Mechanical Engineering** with specialization in "**Machine Design and Analysis**" at National Institute of Technology, Rourkela is an authentic work carried out by her under my supervision and guidance.

To the best of my knowledge the matter embodied in the thesis has not been submitted to any other university/Institute for the award of any degree or diploma.

Place: Rourkela

Date:

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CONTENTS

Dedicated to

My Parents

&

“Seema”

ABSTRACT

Polymeric materials reinforced with synthetic fiber such as glass, carbon and aramid provide advantages of high stiffness and strength to weight ratio and their use is very well justified in varieties of applications. Despite these advantages, the wide spread use of synthetic fiber-reinforced polymer composite is declining because of their higher cost and adverse environmental impact. On the other hand the use of natural fiber to develop environment friendly green materials are attracting researches worldwide due to their advantages like biodegradability, high weight, low-cost and high specific strength compared to synthetic fiber. In this category, silk is also a natural fiber. It is mainly branded as a status symbol of wealth and luxury. Just like any other conventional raw materials in silk manufacturing also wastes are generated during manufacturing. These wastes to some extent are used for manufacturing less delicate products such as sportswear, draperies and upholstery. In order to find value added application of this waste, the present work deals with preparation and characterization of epoxy composite with addition of waste silk fibers.

In this work, the waste silk was collected from the silk industries located in Raigarh, (district) Chattishgarh, India. This waste material in local language is called as “Ghincha”. The composites with different weight fraction of silk fiber (2, 4, 6 and 8 wt %) were prepared with epoxy as matrix material by hand lay-up technique. Experiments were conducted under laboratory condition to assess the effect of different environment such as subzero, steam, saline water and natural conditions for various time lengths. The change in weight, volume and dimensions are studied for various treatments. Shear strength of the composites was evaluated by three point bend test as per ASTM standard. The increase the potential use of waste silk fiber, in the present study the erosion wear behavior of silk fiber and hybrid laminate composites with Jute and synthetic fiber glass have also been carried out.

The composites degradation time varies with different environmental treatments. The erosion response of silk fiber composites shows semi ductile behavior. However the hybridization improves the erosion response of silk fiber from semi ductile to ductile nature.

Hence the above results of environmental effect and erosive response of silk fiber clearly show the potential application for making partition board, false ceiling, doors and window panels.

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SANYUKTA GUPTA

LIST OF FIGURES

LIST OF TABLES

CHAPTER – 1

Introduction

1.1 Background and Motivation

When two or more material with different properties is combined together they form a composite material [1]. The constituents are combined in such a way that they keep their individual physical phases and are non soluble in each other or do not form a new chemical compound. That is why a composite is considered to be any multi phase material system that exhibits a combination of properties that makes the composite superior to each of the constituent phases. This criterion has provided the main motivation for the research and development of composite material worldwide. There are basically two category of constituent material, one constituent is called reinforcing phase and one in which the reinforcing phase is embedded is called matrix. The primary function of matrix is to hold the fiber to form a certain shape. Besides, the functions of the matrix are also to transfer stress between the reinforcing fibers and to protect them from mechanical and environmental damage. The function of reinforcing phase in matrix is to improve the mechanical properties such as strength, stiffness etc. As per Berghezan [2] the composite material is to be designed in such a way that the individual component retain their characteristic are so incorporated that the composite take advantage of their superior properties without compromising on the weakness of either. There are basically three major types of composite materials available designated as per the matrix material used. The matrix material can be metallic, polymeric or can even be ceramic. When the matrix is a polymer, the composite is called polymer matrix composite.

Fiber reinforced polymer (FRP) composite are the most common advanced composites. These composites consist of a polymer matrix reinforced with thin diameter fibers. The reasons why they are the most common composite include low cost, high strength, and simple manufacturing processes. However they are not free from drawbacks. The drawbacks of FRP composite are, low operating temperature, high coefficient of thermal and moisture expansion and low elastic properties in transverse direction. Still many FRP composites offer a combination of both strength and modulus that are either comparable to or better than many traditional materials. There are many polymer resin system used as matrices in FRP composites. They can be classified as thermo plastic (polyethylene, polypropylene, nylon etc) and thermoset (epoxies, polyesters, vinyl ester etc) polymer. Thermoplastic polymer can be repeatedly softened and formed by increasing the temperature or hardened by

decreasing the temperature, while the thermoset polymers are insoluble and infusible after cure.

As far as reinforcement is concerned fibers occupy the largest weight fraction in a FRP composite and it share its major portion of the load that act on the composite structure. The reinforcing fibers can be oriented during fabrication there by giving ample opportunity to the designer to tailor down the properties in specific direction. The major fibers in use today are glass, carbon and aramid. Recently research on engineering interest have been shifting from traditional synthetic fiber composite to lignocellulosic natural fiber composite due to their advantages like high strength to weight ratio, non carcinogenic and biodegradability[3-6]. The term natural fiber covers a broad range of vegetables, animal and mineral fibers. Availability of natural fibers and easy of manufacturing is tempting researcher to try locally available inexpensive natural fibers as reinforcement material in polymer matrix. The other advantages associated with natural fibers are non abrasive nature, low energy consumption, biodegradability, light weight and low cost.

Among various natural fibers silk is also a natural (protein) fiber. Very light weight silk textile materials can be manufactured from silk filaments. The mysterious legend of silk's discovery in China around 3000 BC and its closely guarded cultivation has forever branded it as a status symbol of wealth and luxury, which makes it more than just a textile venture. Silk is one of the oldest textile industries, and even older art form. Silk is primarily made up of proteins, and it is in close composition to human skin (Hyuarinen). Silk is least wasteful and more natural than any of its manmade substitutions. However the waste products sometimes can be numerous when compared to the final product. Most of these products can be reused or can provide another commercial value. The byproduct of manufacturing silk includes the unusable parts of the pupa and cocoon. They can be processed to make dupion silk, or reprocessed into flow-silk and spun silk yarns.

The real source of silk waste is from the cocoons and manufacturing processes, not from the disposal of finished silk product. In order to have complete cocoons from which to unwind the silk threads the pupa must be killed, because if it grew into a moth it would break the cocoon on it emerged, which would ruin it (Fig-1.1). Some commercial uses for silk waste include "matka" which is derived from pure silk waste by hand spinning while skipping the degumming process. "Tusser," another alternate use of silk waste, is derived from the cocoons left over from silk reeling, and is spun into yarn. Another form is called

“floss silk” can be manufactured from any kind of defective cocoon, “but principally it is processed from pierced, end missing, and double cocoons. Floss silk is beneficial as paddy against cold weather and as a basis for hand spun yarns”. The non filament waste materials have also found alternative commercial value. The pupae removed from the cocoons may be used for fertilizer, feed stuff and other agriculture purposes. The waste from spinning silk yarn may also be respun into other forms of yarn and woven into textured fabrics for less delicate uses such as sportswear, draperies and upholstery.

Figure-1.1 Cocoon (Bombix Mori)

Silk fiber is a valuable resource with good properties and it can be used in a great many value added products. Using silk fiber for composite has many advantages. Silk is renewable versatile nonabrasive, porous, hydroscopic, viscoelastic, biodegradable, combustible, computable and reactive. The fiber has a high aspect ratio, high strength to weight ratio, is low in energy conversion, and has good insulation properties. Using silk waste fiber reinforced epoxy composites can be very cost-effective material especially for building & construction industry (panels, false ceilings, partition boards etc.), packing, automobile & railway coach interiors and storage devices.

In order to find an application to this valuable waste, in this present work an attempt has been made to develop a polymer matrix composite (epoxy resin) using silk yarn as reinforcement. Efforts are made to study the mechanical properties and effect of different environment on the flexural properties of the composite. Moisture absorption behavior of the composite have also been studied after subjecting them to different treatments like steam,

saline and subzero conditions. Attempts have also been made to study the erosive wear behavior of the silk fiber reinforced composite. A comparative study on erosive wear behavior has also been made after preparing a hybrid composite of silk waste along with synthetic fiber (glass) and natural fiber (jute). Effect of hybridization and layering sequence effect of jute, silk and glass on tensile, flexural, and interlaminar shear strength have also been studied.

1.2 Thesis Outline

The remainder of this thesis is organized as follows:

Chapter 2: Previous work relevant to the present investigations available in literatures is described in this chapter

Chapter 3: This chapter describes the details of materials required, fabrication techniques and also the effect of environment on mechanical properties and moisture absorption characterization of the composite under investigation.

Chapter 4: In this chapter the erosion wear behavior of the composite is presented.

Chapter 5: This chapter deals with Preparation of the hybrid composite. The effect of hybridization and layering sequence effect on erosive wear behavior.

Chapter 6: Conclusions and recommendations for future work are presented in this chapter

CHAPTER – 2

Literature survey

2. Literature survey:

Literature survey is carried out to get the background information on the issues to be considered in the present research work and to focus the relevance of the present study. The purpose is also to present a thorough understanding of various aspects of natural fiber polymer composite with a special attention to their mechanical properties and erosion wear behavior.

2.1 NATURAL FIBERS: Initiative in Product Development.

In fiber reinforced polymer composites, the fibers can be either synthetic fibers or natural fibers. Natural fibers constituents are mainly of cellulose fibers, consisting of helically wound cellulose micro fibrils, bound together by an amorphous lignin matrix. Lignin keeps the water in fibers; acts as a protection against biological attack and as a stiffener to give stem its resistance against gravity forces and wind. Hemicellulose found in the natural fibers is believed to be a compatibilizer between cellulose and lignin [7].The use of lignocellulosic fibers as reinforcements for polymeric materials has been growing during the last decade or so to replace synthetic fibers, especially glass fibers in composites, for different industrial sectors, such as packaging, automobiles [8, 9] and even in the building sector [10]. This is mainly due to their unique characteristics, such as availability, biodegradability, low density, non-toxic nature, less abrasiveness to plastic processing equipment, useful mechanical properties and low cost [11]. The chemical composition of natural fibers may differ with the growing condition and test methods even for the same kind of fiber. The physical mechanical properties of natural fibers are greatly influenced by their chemical compositions. The properties of some of these fibers are presented in Table-2.1 [12]. It is evident from Table-2.1 that, the tensile strength of glass fiber is substantially higher than that of natural fibers even though the modulus is of the same order. However, when the specific modulus of natural fibers is considered, the natural fibers are better as compared to glass fibers. Therefore, these higher specific properties are the major advantages of natural fiber as reinforcement in polymer composites for weight sensitive applications.

Table 2.1 Properties of natural fibers [12]

Fiber	Tensile strength (MPa)	Young's modulus (GPa)	Elongation at break (%)	Density (g/cm3)
Abaca	400	12	3-10	1.5
Alfa	350	22	5.8	0.89
Bagasse	290	17	-	1.25
Bamboo	140-230	11-17	-	0.6-1.1
Banana	500	12	5.9	1.35
Coir	175	4-6	30	1.2
Cotton	287-597	5.5-12.6	7-8	1.5-1.6
curaua	500-1,150	11.8	3.7-4.3	1.4
palm	97-196	2.5-5.4	2-4.5	1-1.2
Flax	345-1,035	27.6	2.7-3.2	1.5
Hemp	690	70	1.6	1.48
Henequen	500±70	13.2 ± 3.1	4.8 ± 1.1	1.2
Isora	500-600	-	5-6	1.2-1.3
Jute	393-773	26.5	1.5-1.6	1.3
Kenaf	930	53	1.6	-
Nettle	650	38	1.7	-
Oil palm	248	3.2	25	0.7-1.55
Piassava	134-143	1.07-4.59	21.9-7.8	1.4
Pineapple	400-627	1.44	14.5	0.8-1.6
Ramie	560	24.5	2.5	1.5
Sisal	511-635	9.4-22	2.0-2.5	1.5
E-Glass	3400	72	-	2.5

Natural organic fibers can be derived from either animal or plant sources. The majority of useful natural textile fibers are plant derived, with the exceptions of wool and silk. All plant fibers are composed of cellulose, whereas fibers of animal origin consist of proteins. Natural fibers in general can be classified based on their origin, and the plant-based fibers can be further categorized based on part of the plant they are recovered from. An overview of natural fibers is presented in Figure-2.1 [13].

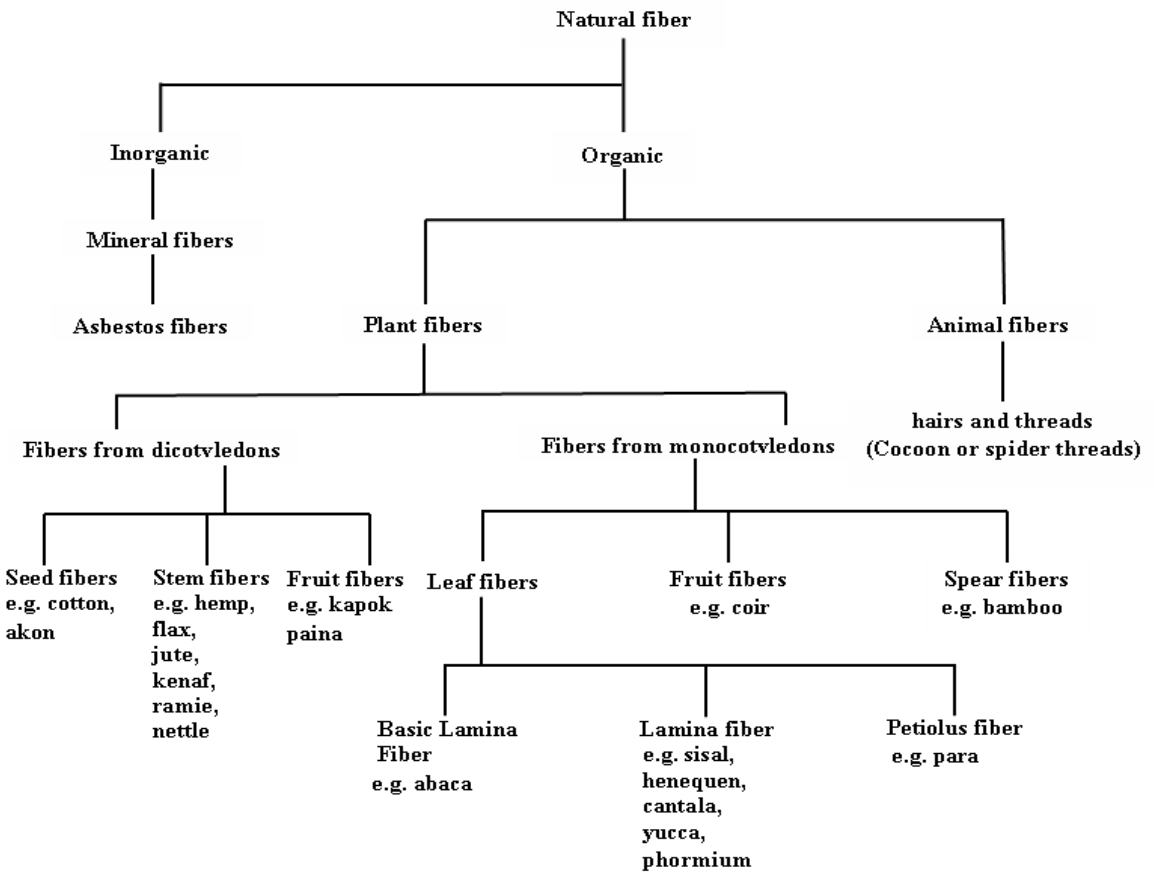

Figure-2.1 Overview of natural fiber

A great deal of work has already been done on the effect of various factors on mechanical behavior of natural fiber reinforced polymer composites. The post-impact behavior of jute fiber reinforced polyester composites subjected to low velocity impact has been studied by Santulli [14]. Effect of fiber content on tensile and flexural properties of pineapple fiber reinforced poly (hydroxybutyrate-co-valerate) resin composites has studied by Luo and Netravali [15].The mechanical behavior of jute and kenaf fiber reinforced polypropylene composites has been studied by Schneider and Karmaker [16]. It is concluded from their study that jute fiber based composites provides better mechanical properties than kenaf fiber based composites. The effect of various loading rate on mechanical properties of jute/glass reinforced epoxy based hybrid composites has studied by Srivastav et al. [17]. The mechanical properties of jute fiber reinforced polyester composites were evaluated by Gowda et al. [18]. It is reported from their study that jute fiber based composites have better strengths as compared to wood based composites. The limited use of natural fiber composites is also connected with some other major disadvantages still associated with these materials. The fibers generally show low ability to adhere to common non-polar matrix materials for

efficient stress transfer. Furthermore, the fibers inherent hydrophilic nature makes them susceptible to water uptake in moist conditions. Natural fiber composites tend to swell considerably with water uptake and as a consequence mechanical properties, such as stiffness and strength, are negatively influenced. However, the natural fiber is not inert. The fiber-matrix adhesion may be improved and the fiber swelling reduced by means of chemical, enzymatic or mechanical modifications [19].

S.k.Acharya et al. [20] studied the weathering behavior of bagasses fiber reinforced polymer composite. Their report states that the shear stress of the composite is very sensitive to the treatments. The shear stress decreases with increasing in fiber weight fraction. Deo et al. [21] investigated that the effect of moisture absorption on mechanical properties of chopped natural fiber reinforced epoxy composite and have reported the same type of behavior. Both the volume and weight change of the composite attains stability after certain period of exposure in different environment the composite subjected to and the flexural strength of composite are very sensitive to the fiber loading.

During the last few years, a series of works have also been done to replace the conventional synthetic fiber with natural fiber composites [19, 22, 23-28]. Nevertheless, certain aspects of natural fiber reinforced composite behavior is still poorly understood such as their visco elastic, visco plastic or time-dependent behavior due to creep and fatigue loadings [29], interfacial adhesion [30, 31], and tribological properties. Little information concerning the tribological performance of natural fiber reinforced composite material [32–35] has been reported. In this context, long plant fibres, like hemp, flax [30, 31], and bamboo [33, 34] have considerable potential in the manufacture of composite materials. Likewise, silk fibers may also have considerable potential as reinforcement for polymer and may provide advantages when used as a substitute for conventional synthetic glass fiber.

Though silk is extensively used as a valuable material for textiles purposes the studies on composites with silk yarn as reinforcement are meager. Padmapriya et al. [36] studied the tensile flexural and chemical resistance and the morphological aspect of silk fabric reinforced epoxy composites. They prepared these laminates with varying content of silk fabric in epoxy. They studied the mechanical properties like tensile and flexural strength of the composites. Their result shows that the tensile and flexural strength properties of the composite increases with increase in silk fabric content. These composites also showed good chemical resistance to some acids, alkalis, and solvents. The interfacial bonding between the

reinforcement and the matrix was also examined using SEM technique. They have reported that the fiber pullout were the predominant mode of failure. In another study [37] they reported the impact and the compression properties of waste silk fiber/epoxy composites with reference to the varying waste silk fabric content within them. They found that maximum strength is observed for the optimum fiber loading. Properties like density, void content, and weight reduction were also determined for these composite. Their report states that there is a marked reduction in the void content of the composites as the weight percentage of the silk increased. The total weight of the composite reduced to a small percentage with an increase in the fabric reinforcement in the composite.

Many researchers have investigated the erosion behavior of various polymers and their composites [38-50]. Harsha et al. [51] reports about the solid particle erosion behavior of randomly oriented short E-glass, carbon fiber and solid lubricants (PTFE, graphite, MoS2) filled polyetherimide (PEI) composites. They reports that Polyetherimide and its glass, carbon fiber reinforced composites showed semi-ductile erosion behavior with peak erosion rate at 60^0 impingement angle. The impact velocity had a pronounced effect on the erosive wear of PEI and its composites. For PEI and other composites, the steady-state erosion rate (E) is related to particle velocity (v) as $E = kv^{n.}$

Zahavi et al. [52] have investigated Solid particle impingement erosion of uncoated composite materials of quartz-polyimide, glass-epoxy and quartz polybutadiene and determine the effect of the mass of sand impacted and the impact angle. Their study reveals that progressive mass loss was observed on all materials as the mass of sand impacted increases, one glass-epoxy composite exhibited erosion which was less than that of the other composites by half an order of magnitude; this is attributed to better adhesion between the matrix and fibers, a higher percentage of fiber loading and lower porosity. This glass-epoxy composite exhibited semi ductile erosion behavior with a maximum weight loss at an impingement angle of 45^0 - 60^0 while the others eroded in a brittle manner with a maximum at an impingement angle of 75^0 - 90^0.

Barkula et al. [53] have studied the erosive wear behavior of glass fiber reinforced thermoplastic polypropylene composites. Their results showed a strong dependence of the erosive wear on the jet angle. The GF/EP systems presented a brittle erosion behavior, with maximum weight loss at 90^0 impact angle. It was established that good fiber/matrix adhesion improved the resistance to erosive wear. On the other hand, the relative fiber orientation had a negligible effect except the erosion at 30^0 impact angle.

Srivastava et al. [54] investigated about the fracture toughness and fracture surface energy of epoxy, epoxy/fly-ash, epoxy/carbon, fibre, epoxy/ carbon fiber/fly-ash, epoxy/ glass fiber and epoxy/glass fiber/fly-ash composites. Their results showed that a fly-ash particle can arrest the crack path and thus improve the fracture properties of fiber reinforced plastic (FRP) composites.

Miyazaki et al. [55] studied about effect of matrix materials, reinforcement fibers, interface strength between matrix material and fibers, impact angle, and particle velocity on the solid particle erosion behavior of short glass carbon fiber reinforced nylon 66 resin, ABS resin .They found that the erosion rate is larger in FRP, that in neat resin and the erosion rate of FRP decreases with the increase of interface strength between matrix material and fibers.

Roy et al. [56] studied about the solid particle erosion behavior of four different types of polymer matrix composites namely glass epoxy resin, glass phenolic resin (modified), glass phenolic resin (unmodified) and glass polyester resin were used. Their result showed that the glass-reinforced epoxy resin composite exhibits the lowest erosion rate and glass-reinforced phenolic resin (modified) shows the highest erosion rate (at $\alpha=30^0$ and 90^0 for V=38 and 45 m/s). The erosion rates of glass-polyester resin and glass- (unmodified) phenolic resin exhibit intermediate values. Composites having thermoset matrix (epoxy and phenolic) behave in a brittle way while the composites with thermoplastic matrix (polyester) respond in a ductile manner.

To enhance the suitability of natural fiber in different applications recently some researchers tried to incorporate synthetic fiber into natural fiber

Ahmeda et al. [57] Studied the Effect of stacking sequence on tensile, flexural and interlaminar shear properties of woven jute-glass fabric reinforced isothalic polyester composites and reported that, Incorporation of glass in jute fibre composites enhances the properties of resulting hybrid composites and the Layering sequence (altering the position of glass plies) significantly affects the flexural and inter laminar shear strength

Santulli et al. [58] studied the comparison between two composite architectures namely a hemp/epoxy random mat and a jute/epoxy plain weave laminate, both with 45±2% volume and their work reported that manufacturing a hybrid laminate, using jute / epoxy plain woven and hemp / epoxy random mat, most preferably the latter (inherently

stronger) as skins and the former as core, would be able to reduce the scattering in impact resistance values and lead to a better predictability of its impact behavior.

2.2 Summary of the present work.

The literature survey presents above reveals the following.

Much work has been done on a wide variety of natural fibers combining with polymer matrices resulting improvement in mechanical properties of the composites compared with the matrix material.

The major disadvantage which makes natural fiber less attractive is the poor resistance to moisture absorption. Accordingly numbers of research efforts have been developed to understand the mechanism and methods to lower down this rate of moisture absorption.

Studies have been carried out worldwide on erosion wear behavior of various fiber reinforced composite. However silk fiber reinforced composite erosion studies has not been carried out so far.

To take the positive aspect combination of both natural and synthetic fiber studies on mechanical strength has been carried out to some extent. However the tribological behavior of this type of combination has not been touched so far as per the above survey.

2.3 Objective of the present work.

The objective of the present work:

- To fabricate silk fiber composite with different fiber weight fraction.
- To study the effect of different environment on the tensile and flexural properties of the composite.
- Study of solid particle erosion behavior of the composite.
- To fabricate layered composite with silk, jute and glass fiber.
- Evaluation of mechanical properties such as tensile strength, flexural strength, interlaminar shear stress and micro hardness of different layered composite.
- Study of solid particle erosion behavior of layered composite.

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CHAPTER – 3

MATERIALS AND METHOD

3.1 Raw Materials:

Raw materials used in this experimental work are listed below

1. Natural fiber (waste silk)

2. Epoxy resin

3. Hardener

3.1.1 Waste Silk Fiber:

Silk is one of the oldest textile industries, and even older art form. Silk is primarily made up of proteins, and it is in close composition to human skin (Hyuarinen).

The short lengths of silk obtained from silk waste is “spun-silk”. It can be obtained from upholestry cocoons, double cocoons, the floss brushed from cocoons before reeling; the coarse and uneven silk friezes at the ends of each cocoon, or the machine scrap left over from reeling. These “inferior silk filaments are combed and spun together as silk thread”. Spun silk soft less expensive, less lustrous, strong and elastic as reeled silk. Because it uses shorter filaments, spun silk will become fuzz easier. The waste from spinning silk yarn may also be respun into other forms of yarn and woven into textured fabrics for less delicate uses such as sportswear, draperies and chopped Tussah Silk silver is used for soap making and as fiber for embellister. It is natural honey colour as shown in Figure-3.1.

Silk is itself a relatively clean substance whose manufacturing requires relatively little energy and superfluous resources. The finished products of pure silk fabrics are organic and biodegradable. The bulk of silk waste comes from manufacturing, and it is in this stage where economic losses can be cut by finding commercial value in any by-products. Almost all forms of silk waste in this stage can be used for other fibers, many of which are similar to pure silk quality.

Figure-3.1 Silk fiber wastes

In the present work waste silk fibers of 2, 4, 6 and 8 weight percent have been taken as reinforcement in the polymer matrix.

3.1.2 Epoxy Resin:

The type of epoxy resin used in the present investigation is araldite LY556 which is chemically belongs to epoxide family. Its common name is BisPhinol-A-Diglycidyl-Ether. It is supplied by CIBA GUGYE India Limited.

3.1.3 Hardener

The hardener with IUPAC name NNO-bis (2aminoethylethane-1,2diamin) has been used with epoxy designated as HY951. This has a viscosity of 10-20 MPa at 25ºc.

3.2 PREPARATION OF THE COMPOSITE:

The following procedure has been adopted for preparation of the specimen.

3.2.1 Fiber Preparation:-

The waste silk fibers used in present study were collected from silk industries located in Raigarh (Chhattisgarh) called GHINCHA in local language. The silk threads were entangled and so they were woven with hand to separate long threads. Then the foreign particles present in the fiber were separated by hands and they were washed by high pressure air to remove the undesirable foreign matter.

3.2.2 Composite Preparation:-

Per-pex sheet mould (dimension 130X100X6mm) Figure-3.3(a) was used for casting the composite sheet. A mold release spray was applied at the inner surface of the mold for quick and easy release of the composite sheet. A calculated amount of epoxy resin and hardener (ratio of 10:1 by weight) was thoroughly mixed with gentle stirring to minimize air entrapment. After keeping the mold on a glass sheet (coated with wax) a thin layer (≈ 2mm thickness of the mixture was poured in the mold. The retting fiber bundles were then separated and made like mats. The remainder of the mixture was then poured into the mold. Care was taken to avoid formation of air bubbles. Pressure was then applied from the top and the mold was allowed to cure at room temperature for 72 hrs. During application of pressure some amount of epoxy and hardener squeezes out. Care has been taken to consider this loss during manufacturing so that a constant thickness of sample could be manufactured. This procedure was adopted for preparation of 2, 4, 6 and 8 % weight fractions of fiber. After 72 hrs the samples were taken out of the mold, cut into different sizes and kept in air tight container for further experimentation. Figure-3.2(b) and(c) shows the photograph of the samples cut from the slab.

Figure- 3.2 (a) Mold **(b) Specimen for Tensile test** **(c) Flexural Test**

3.3 EXPERIMENTAL PROCEDURE:

To find out the effect of environment on mechanical properties the composite samples were subjected to various treatments like:

a. Steam treatment
b. Saline water treatment
c. Subzero condition

In each condition a set of composite (2, 4, 6 and 8% wt percent) were tested for various time lengths. Steam treatment was conducted at 100^0C with 95% relative humidity. Saline water treatment was done with 5% concentration at room temperature. Subzero treatment was conducted at -23^0C. At the end of the treatment at each condition the dimension and weight change were measured.

3.4 CHARACTERIZATION

3.4.1 Density

The theoretical density of composite materials in terms of weight fraction is found out from the following equations as given by Agarwal and Broutman [59].

$$\rho_{ct} = \frac{1}{\left(\frac{W_f}{\rho_f}\right) + \left(\frac{W_m}{\rho_m}\right)} \tag{3.1}$$

Where '*W*' and 'ρ' represents the weight and density respectively. The suffix *f, m* and *ct* stand for the fiber, matrix and the composite materials. The results are tabulated in Table -3.1

Table-3.1

Density of different Samples

Sample	**Density(gm/cm^3)**
2%	1.1298
4%	1.1382
6%	1.141
8%	1.150

3.4.2 Hardness

Micro-hardness measurement is done using a Lecco Vickers Hardness (LV 700) tester. A diamond indenter, in the form of a right pyramid with a square base and an angle 136^0 between opposite faces, is forced into the material under a load F. The two diagonals X and Y of the indentation left on the surface of the material after removal of the load are measured and their arithmetic mean L is calculated. In the present study, the load considered F = 10 N and Vickers hardness number is calculated using the following equation:

$$H_v = \frac{0.1889F}{L^2} \qquad \text{and} \qquad L = \frac{X+Y}{2} \tag{3.2}$$

Where F is the applied load (N), L is the diagonal of square impression (mm), X is the horizontal length (mm) and Y is the vertical length (mm). The results are tabulated in Table-3.2.

Table-3.2

Micro hardness of different Samples

Sample	Micro-hardness
2%	16.271
4%	17.732
6%	20.746
8%	20.692

3.4.3 Measurement of dimensional change

From the experimental results, dimensional changes (volume and weight) of the composites in each case were measured by using digital calipers for different weathering conditions.

3.4.4 Moisture Absorption Behavior

The weight of the composite increases with increase of moisture content in the specimen. This moisture content in the sample is directly proportional to the duration of exposure into a particular environmental condition.

3.4.5 Mechanical properties

The mechanical properties viz. stress, strain behavior of the composites was evaluated after various treatments. Along with tensile test the samples were tested using three point bend test method from which flexural strength and inter laminar shear stress were found out.

3.5 CALCULATION

(i) Change in dimensions:-

Initial volume was calculated for each composite. During the experimentation after every 8 hours, change in volume was calculated by taking out the samples from the environment they were subjected to. Cumulative volume change was found out after each test. The results are tabulated in Table -3.3 to 3.5.

(ii) Moisture Absorption:-

Moisture absorption was conducted in accordance with ASTM D570-98. Three specimens for each composite system were cut with dimensions of 150x20x5 (length x width x thickness) and the experiment was performed using test samples. The specimens prior to testing were dried in an oven at 80^0 C and then were allowed to cool to room temperature and kept in a desiccator. The weight of the samples were taken before subjected to steam, saline water and sub-zero temperature environments. After expose for 10 hr, the specimens were taken out from the moist environment and all surface moisture was removed with a clean dry cloth or tissue paper. The specimens were reweighed to the nearest 0.001 mg within 1 min of removing them from the environment chamber. The specimens were weighed regularly from 8-64 hrs with a gap of 8 hrs of exposure. The moisture absorption was calculated by the weight difference. The percentage weight gain of the samples was measured at different time intervals by using the following equation:

$$\%M = \frac{(W_f - W_i) \times 100}{W_i} \qquad (3.3)$$

where 'W_i' and 'W_f' denote the initial weight (oven-dry weight) and finale weight after time't', respectively. Equilibrium Moisture Content (EMC) of the sample is the moisture content when the periodic weight change of the sample was less than 0.1% and thus the equilibrium state was assumed to be reached. The results are tabulated in Table -3.6 to 3.8.

(iii) **Tensile Strength**

The tension test is generally performed on flat specimens. The most commonly used specimen geometries are the dog-bone specimen Figure-3.4 and straight-sided specimen with end tabs. The standard test method as per ASTM D3039-76 has been used; length of the test specimen used is 125 mm. The tensile test is performed in universal testing machine INSTRON H10KS .The test were performed with a cross head speed of 10mm/min. For each test composite of five samples were tested and average value was taken for analysis. Figure 3.5 shows the Machine used for the test and the sample in loading condition. The results obtained from the tests is presented in Table-3.9

(iv) Flexural Strength:-

The composite after treated in various weathering conditions, the three point bend test was carried out in UTM 201 machine in accordance with ASTM D2344-84 to measure the flexural strength of the composites. The loading arrangement for the specimen and the photograph of the machine used are shown in Figure-3.6(a) and (b) respectively. The entire specimens were of rectangular cross section of (150x20x5) mm. A span of 120 mm was used maintaining. Specimens of 150mm length and 20mm wide were cut and were loaded in three points bending with a recommended span to depth ratio of 16:1 as shown. The test was conducted on the same machine used for tensile testing using a load cell of 10kN at 2mm/min rate of loading. The flexural stress in a three point bending test is found out by using equation (3.4).

$$\sigma m = \frac{(3f)}{(4bt)} \qquad (3.4)$$

Where σm is the ILSS, f is the load, b is the width and t is the thickness of the specimen under test. The maximum tensile stress was found out from the equation (3.5).

$$Tm = \frac{(3fl)}{(2bt^2)} \qquad (3.5)$$

Where Tm is the tensile stress and l is the gauge length.

Table-3.3

Cumulative Volume Change for 2%, 4%, 6% and 8% fiber weight fraction composite in Steam Treatment

Weight percent of of fiber	2 %			4%			6%			8%		
Treatment (hrs)	Initial volume (mm^3)	Final volume (mm^3)	Diff (mm^3)	Initial volume (mm^3)	Final volume (mm^3)	Diff (mm^3)	Initial volume (mm^3)	Final volume (mm^3)	Diff (mm^3)	Initial volume (mm^3)	Final volume (mm^3)	Diff (mm^3)
0	6289.92	6289.92	0	9415	9415	0	14539.04	14539.04	0	15132.23	15132.23	0
8	6289.92	6583.93	294.01	9415	9762.13	347.13	14539.04	15452.3	913.26	15132.23	16516.7	1384.47
16	6289.92	7502.58	1212.66	9415	10328.26	913.26	14539.04	15833.57	1294.53	15132.23	16712.41	1580.18
24	6289.92	9393.08	3103.16	9415	10961.91	1546.91	14539.04	16404.28	1865.24	15132.23	16655.27	1523.04
32	6289.92	9846.25	3556.33	9415	11311.9	1896.9	14539.04	16642.49	2103.45	15132.23	16670.32	1538.09
40	6289.92	9640.12	3350.19	9415	11461.04	2046.04	14539.04	16405.38	1866.34	15132.23	17274.07	2141.84
48	6289.92	9973.75	3683.83	9415	11350.74	1935.74	14539.04	16633.79	2094.75	15132.23	17280.16	2147.93
56	6289.92	9516.57	3226.65	9415	11519.33	2104.33	14539.04	16633.79	2094.75	15132.23	17288.61	2156.38
64	6289.92	9491.82	3201.91	9415	11678.72	2263.72	14539.04	16802.76	2263.72	15132.23	17228.21	2095.98

Table-3.4

Cumulative Volume Change for 2%, 4%, 6% and 8% fiber weight fraction composite in Saline Treatment

Weight percent of of fiber	2 %			4%			6%			8%		
Treatment (hrs)	Initial volume (mm^3)	Final volume (mm^3)	Diff (mm^3)	Initial volume (mm^3)	Final volume (mm^3)	Diff (mm^3)	Initial volume (mm^3)	Final volume (mm^3)	Diff (mm^3)	Initial volume (mm^3)	Final volume (mm^3)	Diff (mm^3)
0	8052.66	8052.66	0	10234.37	10234.37	0	13657.11	13657.11	0	15501.98	15501.98	0
8	8052.66	8763.79	711.13	10234.37	10987.54	753.17	13657.11	14435.3	778.19	15501.98	15898.67	396.69
16	8052.66	9098.37	1045.71	10234.37	11131.72	897.35	13657.11	14655.36	998.25	15501.98	15878.04	376.06
24	8052.66	9367.58	1314.92	10234.37	11353.22	1118.85	13657.11	14022.6	365.49	15501.98	15586.57	84.59
32	8052.66	9532.09	1479.43	10234.37	11259.79	1025.42	13657.11	14355.47	698.36	15501.98	15668.33	166.35
40	8052.66	9299.81	1247.15	10234.37	11024.06	789.69	13657.11	14086.3	429.19	15501.98	15876.55	374.57
48	8052.66	9159.8	1107.14	10234.37	10891.73	657.36	13657.11	14188.38	531.27	15501.98	15816.43	314.45
56	8052.66	9092.45	1039.79	10234.37	10772.71	538.34	13657.11	14101.75	444.64	15501.98	17298.62	1796.64
64	8052.66	9642.65	1589.99	10234.37	11075.72	841.35	13657.11	13681	23.89	15501.98	17278.42	1776.44

Table-3.5

Cumulative Volume Change for 2%, 4%, 6% and 8% fiber weight fraction composite in Sub-Zero Treatment

Weight percent of fiber	2 %			4%			6%			8%		
Treatment (hrs)	Initial volume (mm^3)	Final volume (mm^3)	Diff (mm^3)	Initial volume (mm^3)	Final volume (mm^3)	Diff (mm^3)	Initial volume (mm^3)	Final volume (mm^3)	Diff (mm^3)	Initial volume (mm^3)	Final volume (mm^3)	Diff (mm^3)
0	11235.71	11235.71	0	12191.15	12191.15	0	10535.84	10535.84	0	13569.21	13569.21	0
8	11235.71	12098.34	862.63	12191.15	12790.52	599.37	10535.84	11418.75	882.91	13569.21	14027.52	458.31
16	11235.71	12179.4	943.69	12191.15	12780.75	589.6	10535.84	11202.26	666.42	13569.21	14347.15	777.94
24	11235.71	12439.71	1204	12191.15	12720.44	529.29	10535.84	10733.45	197.61	13569.21	14416.59	847.38
32	11235.71	12086.03	850.32	12191.15	12820.77	629.62	10535.84	11023.84	488	13569.21	14551.15	981.94
40	11235.71	12262.41	1026.7	12191.15	12816.87	625.72	10535.84	11025.95	490.11	13569.21	14218.83	649.62
48	11235.71	12469.01	1233.3	12191.15	12848.71	657.56	10535.84	11138.53	602.69	13569.21	14315.05	745.84
56	11235.71	12658.51	1422.8	12191.15	12877.55	686.4	10535.84	11165.21	629.37	13569.21	14258.36	689.15
64	11235.71	12538.71	1303	12191.15	12978.82	787.67	10535.84	11025.98	490.14	13569.21	14358.23	789.02

Table-3.6

Variation of moisture absorption data for different fiber weight fraction under steam environment.

Time (Hour)	2%	4%	6%	8%
8	2.52	2.31	2.55	2.85
16	2.74	2.55	2.81	3.25
24	3.89	3.27	3.27	3.71
32	4.1	4.62	3.79	3.88
40	4.56	5	4.9	4.8
48	4.56	5.1	4.91	4.85
56	4.57	5.13	4.95	4.9
64	4.58	5.15	4.98	4.92

Table-3.7

Variation of moisture absorption data for different fiber weight fraction under saline environment.

Time (Hour)	2%	4%	6%	8%
8	1.51	1.02	0.91	0.95
16	1.64	1.11	1.19	1.12
24	1.89	1.5	1.8	1.36
32	2.25	1.9	1.95	1.7
40	2.4	2.4	2	1.9
48	2.65	2.74	2.3	1.95
56	2.68	2.78	2.34	1.95
64	2.7	2.78	2.34	1.95

Table-3.8

Variation of moisture absorption data for different weight fraction under subzero environment

Time (Hour)	Weight fraction of reinforced of fiber			
	2%	4%	6%	8%
8	2.13	1.8	1.48	1.39
16	2.2	2.06	1.76	1.64
24	2.3	2.15	1.95	1.64
32	2.32	2.28	1.95	1.64
40	2.35	2.3	2	1.66
48	2.48	2.45	2.04	1.77
56	2.48	2.48	2.05	1.8
64	2.48	2.52	2.09	1.82

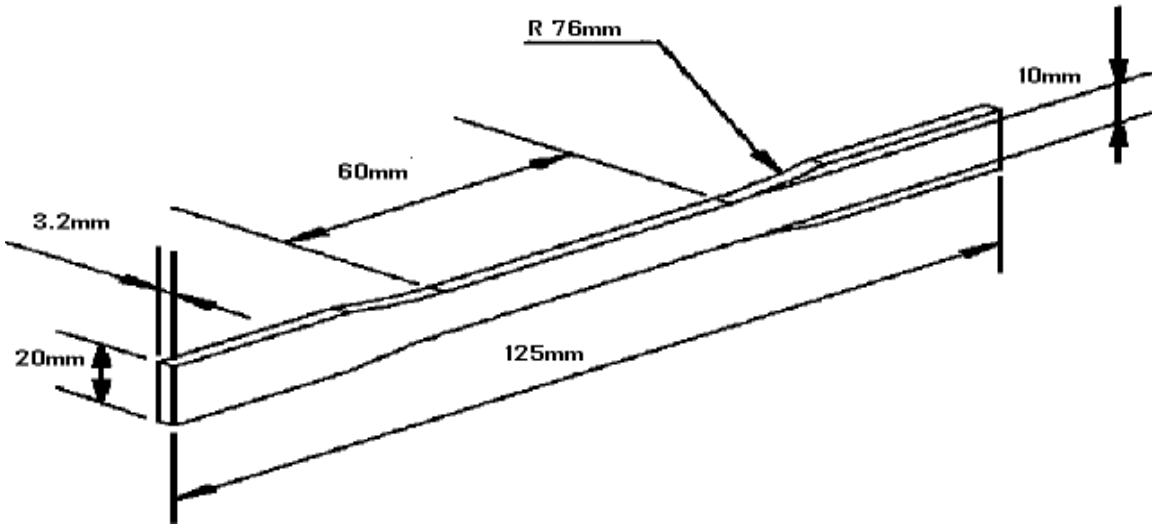


Figure-3.4 Tensile specimen

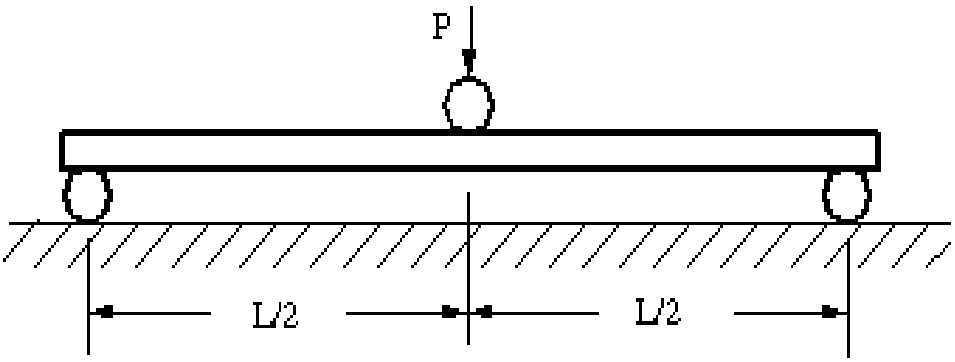
Figure-3.5 INSTRON H10KS TESTING MACHINE

Table-3.9

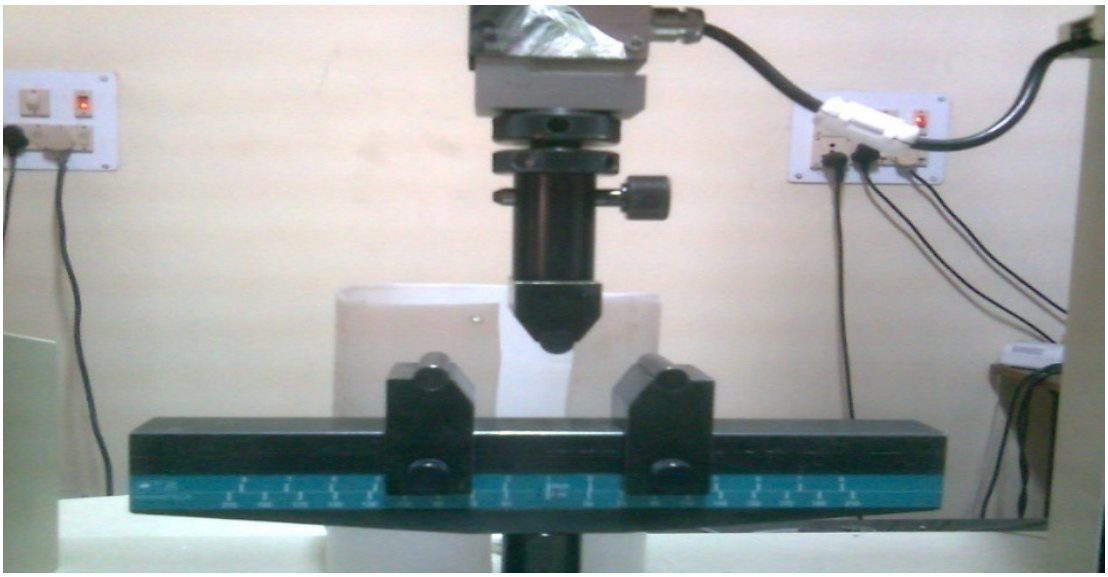
Tensile Stress and Tensile Modulus of composites

Weight percent of fiber	Tensile Stress (MPa)	Tensile Modulus (GPa)
2%	57.35	3.37
4%	69.23	4.07
6%	78.44	4.62
8%	78.38	4.52

The composite specimens of dimensions (150x60x5) mm were cut from the rectangular slabs of the composites. After exposing the composites to various environmental conditions viz. steam, saline and subzero treatments, the changes in the different properties are evaluated. The results obtained from the tests is presented in Table-3.10



(a)



(b)

Figure-3.6 Flexural specimen loading position

Table-3.10

Flexural properties at different environment

weight fraction of fibers (%)	Treatment condition	Flexural Strength (MPa)	Maximum Load (N)
2%	Normal	30.33	67.4
	Steam	28.71	63.8
	Saline	33.44	74.3
	Sub-zero	39.65	88.1
4%	Normal	89.19	198.2
	Steam	102.20	227.1
	Saline	108.81	241.8
	Sub-zero	96.03	213.4
6%	Normal	83.7	186
	Steam	94.37	209.7
	Saline	96.98	215.5
	Sub-zero	91.04	202.3
8%	Normal	68.76	152.8
	Steam	79.97	177.7
	Saline	80.60	179.1
	Sub-zero	72.77	161.7

3.6 RESULT AND DISCUSSION

Figure-3.7 to 3.9 shows the cumulative volume change for different weight fraction of reinforcement subjected to steam, saline and subzero treatment.

Figure-3.7 shows volume change of composite subjected to steam. It is seen from the plot that changes in volume for 8% composite is minimum. All these curves show similar trends with variation in magnitudes. Initially the change in volume increases for all the composites. Beyond certain time of exposure about 56 hrs the change in weight of 2, 4 and 6% composites got stabilized where as in 8% composite the stabilization observes after 40 hrs. This may be due to swelling of the fibers. Since the fibers are tightly packed (exposed area) in 8% reinforcement. They are not getting chance to swell more which results in less weight change.

Figure-3.8 shows weight change of the composites subjected to saline water. Here also the same trend is observed with the difference that for 2, 4 and 6% reinforced composites saturation was observed at about 48 hrs of exposure whereas for 8% reinforced composite the saturation was achieved after 30 hrs of exposure. The rate of swelling in this case might have affected because of accumulation of NaCl ions in the fiber's surface immersed in saline water, which increases with time and prevent swelling of the composite [60].

Figure-3.9 shows the change in volume under subzero treatment for the composite. Linearity in the curves is achieved for 6 and 8% reinforced only after 40 and 24 hrs of treatment. But for 2 and 4% reinforced the linearity was achieved only after 56 hrs of treatment. This type of dramatic shifting of linearity might have happen in sub zero treatment due to intermolecular hydrogen bonding. There is little difference in linearity was achieved at a very early stage for 6 and 8% volume fraction reinforced composite because of the spongy nature of the fiber which absorbs more water.

Figure-3.10 to 3.12 shows the moisture absorption with respect to immersion time in different environments. The moisture absorption increases with immersion time, and got saturated after certain time period. Time to reach the saturation point is not same for all the environments. The saturation time is approximately 40 hrs for steam, and 48 hrs for saline water and 30hrs for sub-zero condition. This shows that environmental conditions play a significant role in moisture absorption process. Figure-3.13 shows maximum moisture absorption of the composite in all the three environments. It is clear from the figure that moisture absorption is maximum in steam condition and minimum in subzero condition irrespective of fiber content. It can be concluded that higher temperature in case of steam environment seem to accelerate the moisture up take behavior. The absorption rate in case of saline water is less than that of steam.

Figure-3.14 to 3.17 shows the variation in flexural strength for the composite in natural, steam, saline and subzero environment. The plot shows that, the samples with 4% reinforced possessed the maximum strength for all conditions. It is observed that when the percentage of reinforced increased from 2 to 4 percentage the flexural strength increased to a very high value, therefore it can be concluded that 4 percentage optimum value of reinforcement for the flexural strength is considered.

3.7 FACTOGRAPHIC ANALYSIS

The fractured surfaces of the samples impregnated with silk yarn as with and without environmental treatments are shown in Figure-3.18(a) to 3.18(d).

The fractured surface of the composite without any environmental treatment is shown in Figure-3.18(a). Matrix cracking is clearly visible on the surface of the composite these cracks are formed on the surface because of initial loading condition. However after the breaking point long extruded fiber that comes out tearing the matrix is clearly visible. The tearing of fibers along the length directions is also seen. When the composite is subjected to steam treated Figure-3.18(b) up on the failure fiber pull out are seen on the fractured surface but are not detached from the matrix surface. Also the fibers pull out shows good wetting of the fiber and polymer.

When the composite is subjected to subzero environment Figure3.18(c), shows altogether a different morphology. The detachment of the fiber from the matrix in the form of fiber pull out is clearly visible. However there is no sign of fiber cracking. In this case, energy is dissipated by shear. At higher fiber loading more fiber surface contributes to energy dissipation, thus improves the fracture resistance appreciable.

Fractured surface of the samples subjected to saline water is shown in Figure-3.18(d) matrix cracking and debonding of the fibers are clearly visible. This also shows the tearing of the fibers along the length direction. Due to cracking of matrix the debris that are formed are distributed and remains on the composite surface. The debrises that are formed might be due to reaction of matrix material with NaCl which is responsible for fiber tearing and separation among the fiber-fiber surface

3.8 CONCLUSIONS

The following conclusions are drawn from the present investigation.

1. Waste silk yarn can successfully be utilized to manufacture composites with low cost but high value product there by providing increased profit for the silk industry.
2. The volume and weight change of the composite attain stability after certain period of exposure.
3. The shear stress of the composite is very sensitive to the treatments. The shear stress decreases with increase in fiber weight fraction.
4. The least swelling is observed with the composite subjected to saline treatment.
5. From the SEM studies it is clear that fiber pullout were the predominant mode of failure.
6. It is also observed that some of the fibers were broken instead of a pullout. Hence the bonding between the silk fibers and the matrix was found to be good.

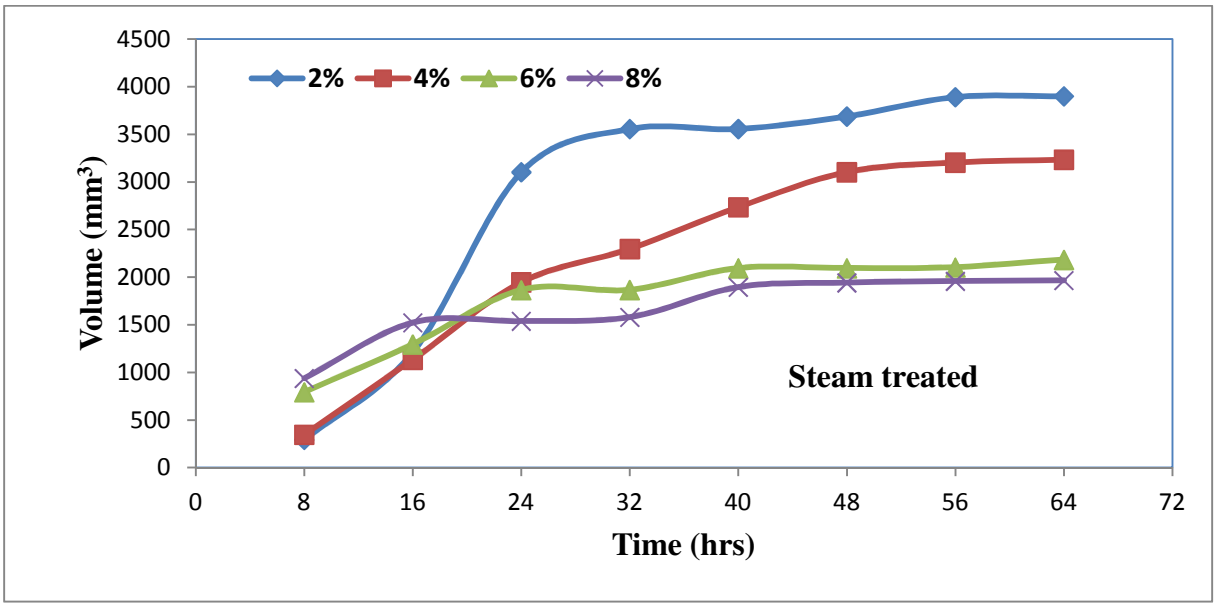


Figure-3.7 Cumulative volume change in different weight fraction of composites for different time of exposure under steam treatment

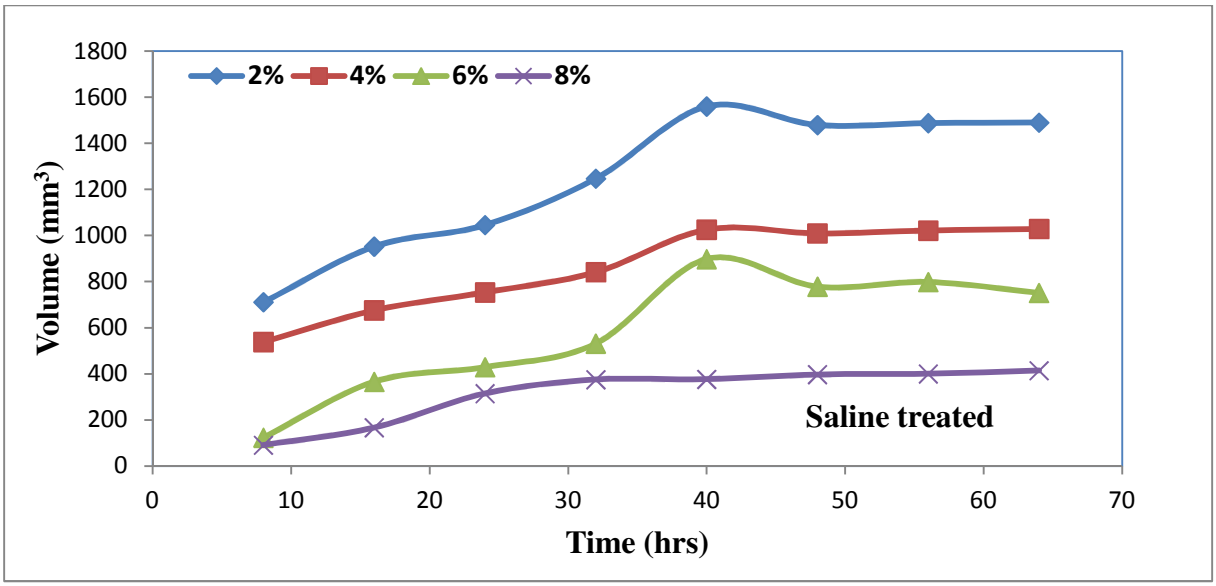


Figure-3.8 Cumulative volume change in different weight fraction of composites for different time of exposure under saline treatment

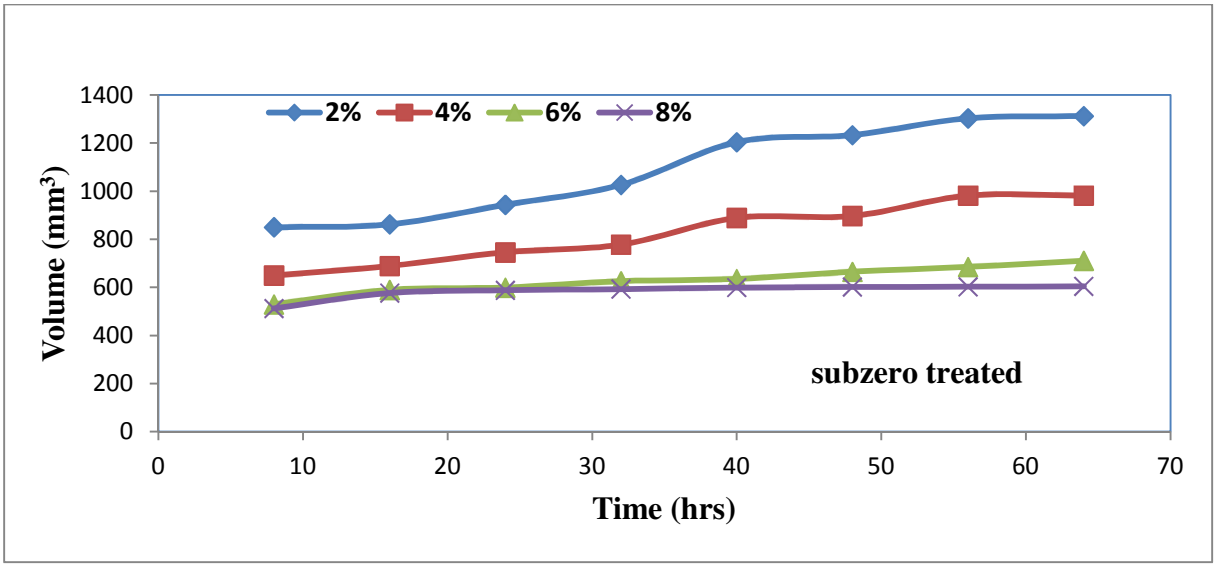


Figure-3.9 Cumulative volume change in different weight fraction of composites for different time of exposure under subzero treatment

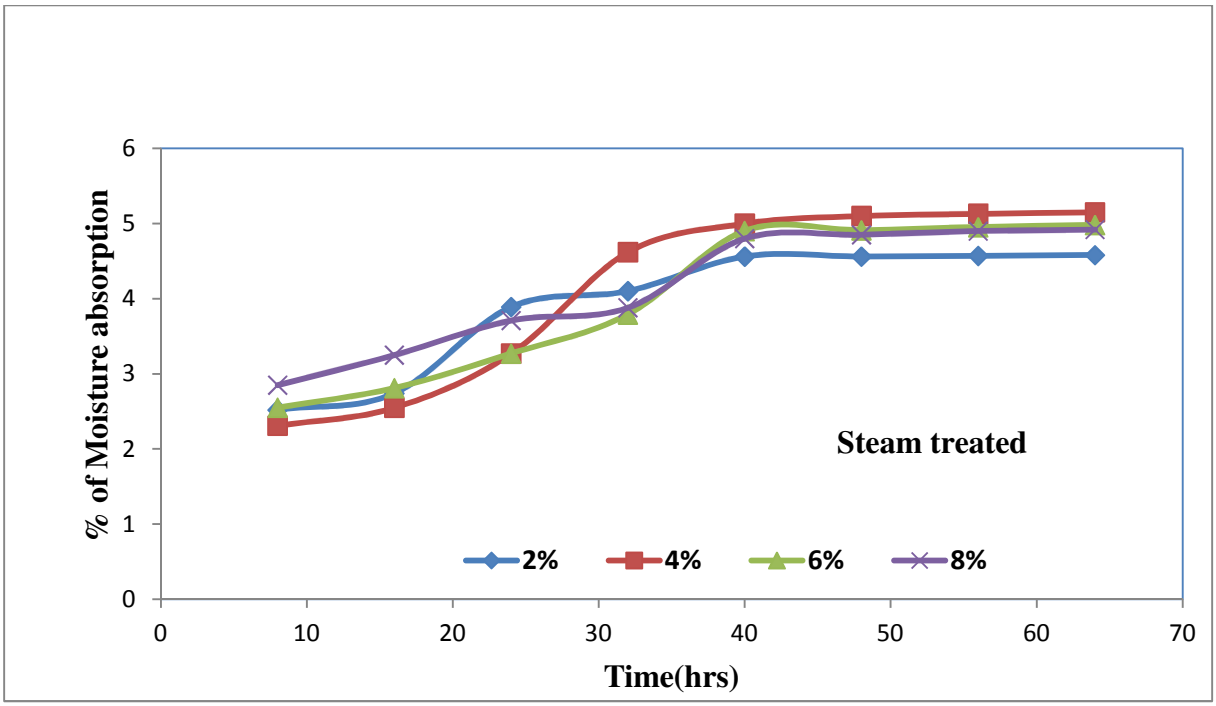


Figure-3.10 Variation of moisture absorption with respect to immersion time in steam treatment

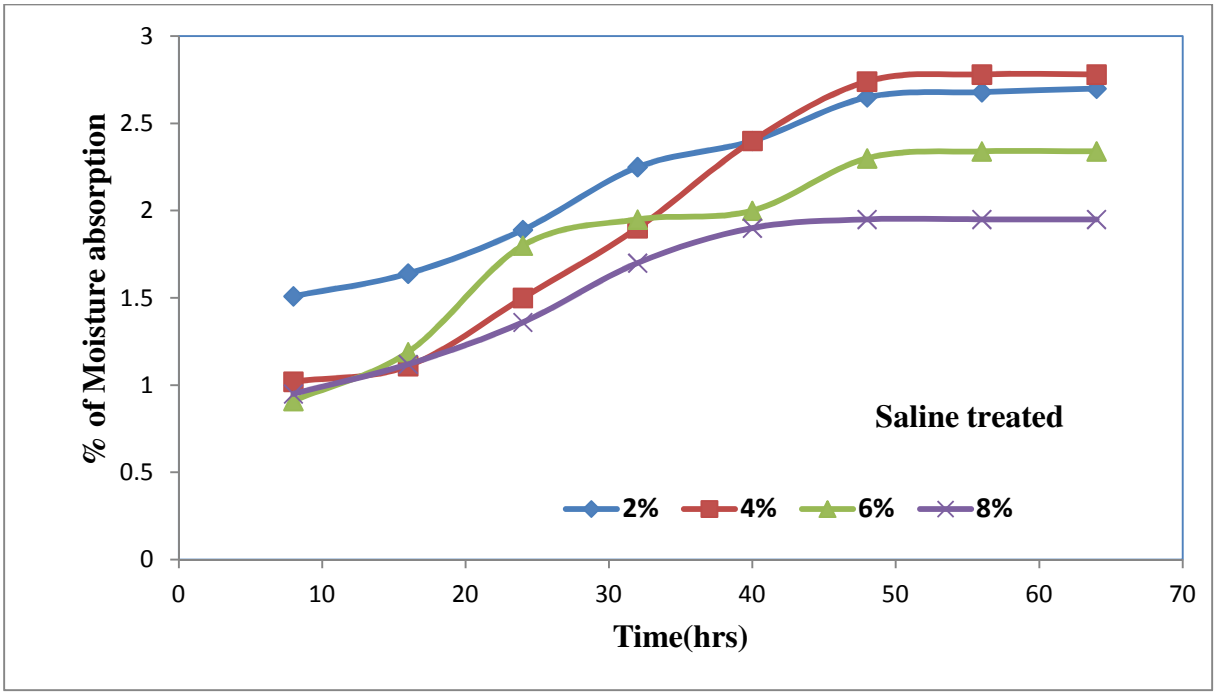


Figure-3.11 Variation of moisture absorption with respect to immersion time in saline treatment

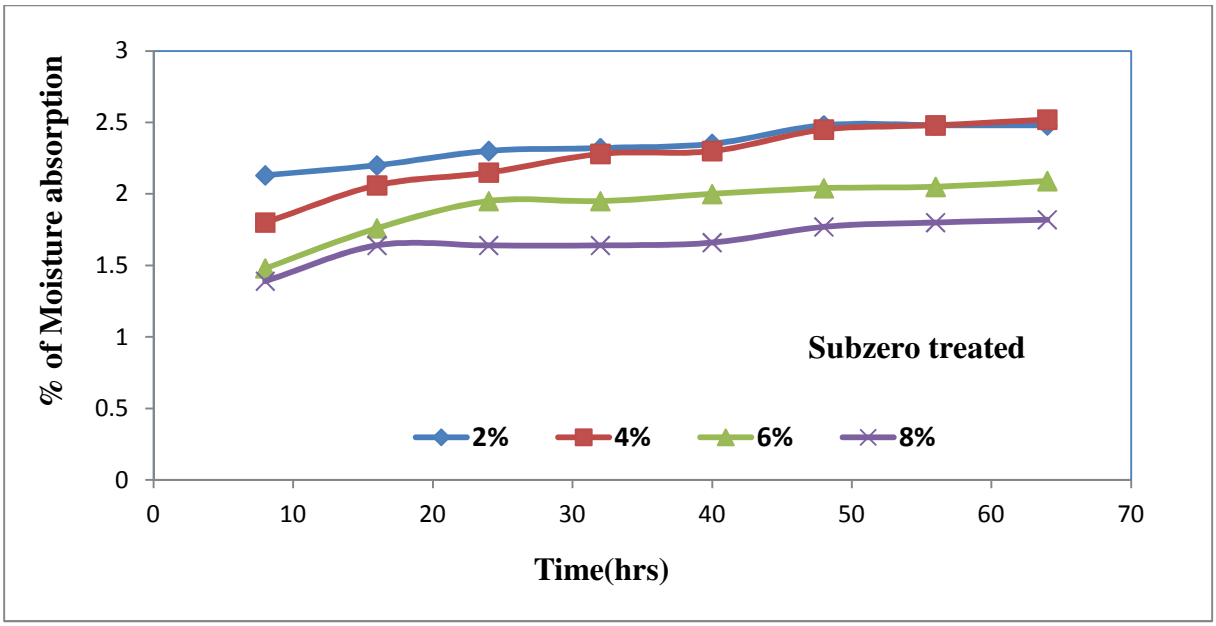


Figure-3.12 Variation of moisture absorption with respect to immersion time in subzero treatment

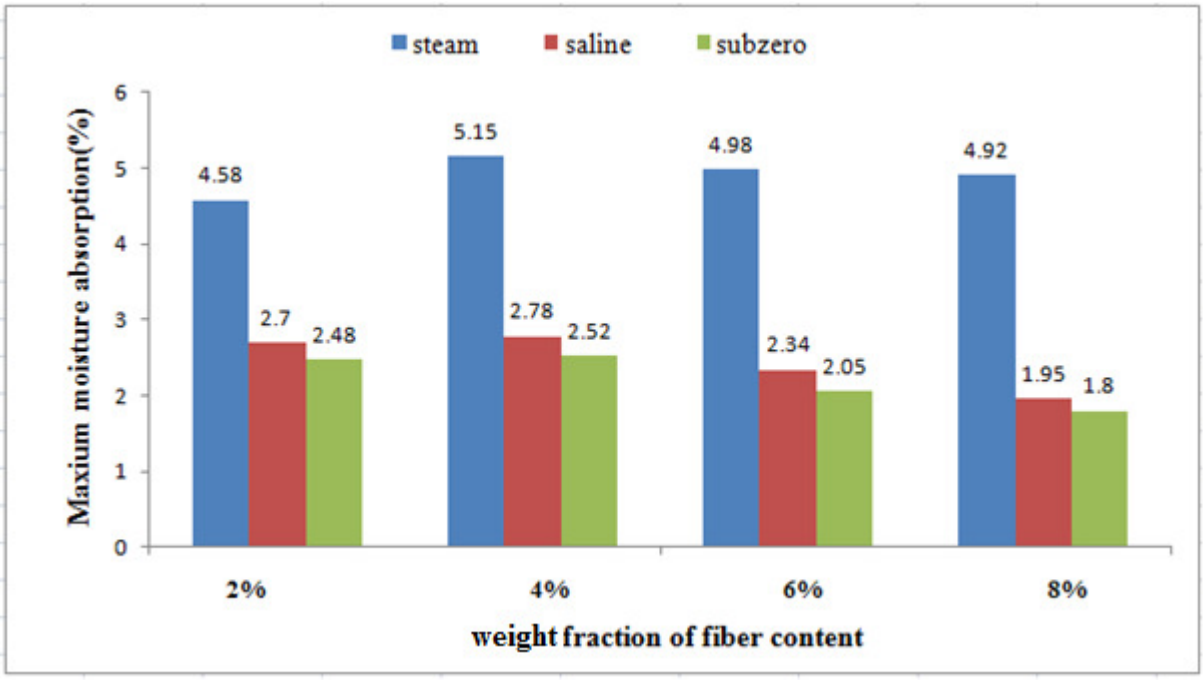


Figure-3.13 Maximum moisture absorption of Waste silk fiber epoxy composite versus fiber loading in all the three environments

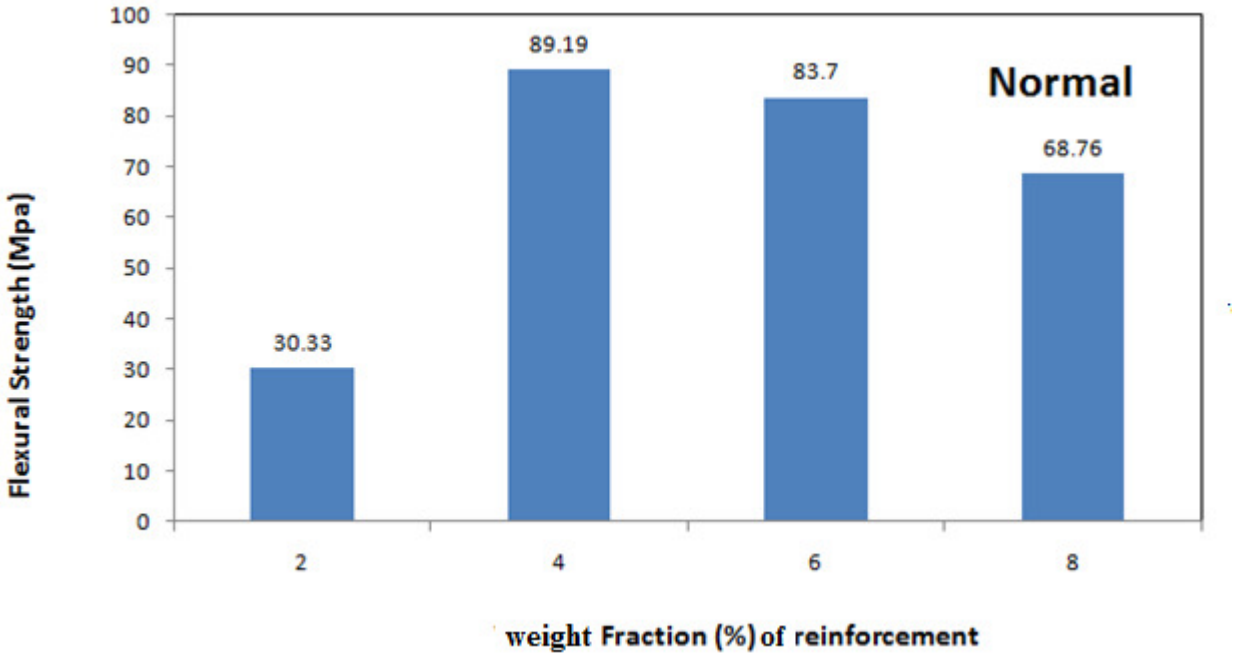


Figure-3.14 Variation of Flexural Strength of different weight fraction without environmental treatment

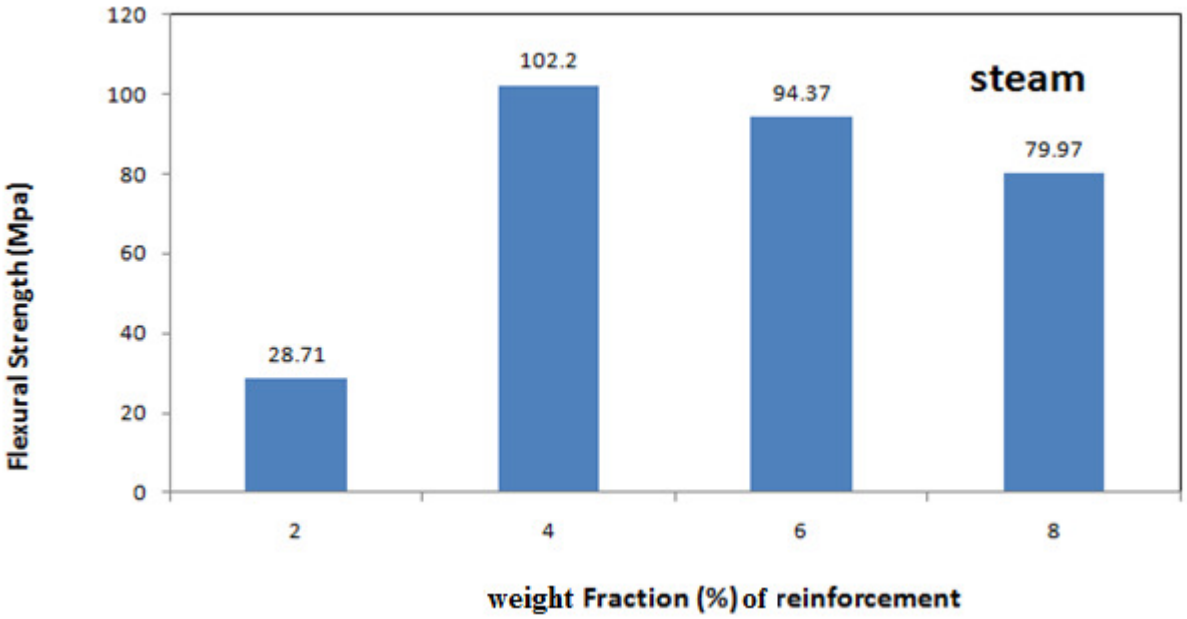


Figure-3.15 Variation of Flexural Strength of different weight fraction in steam environment

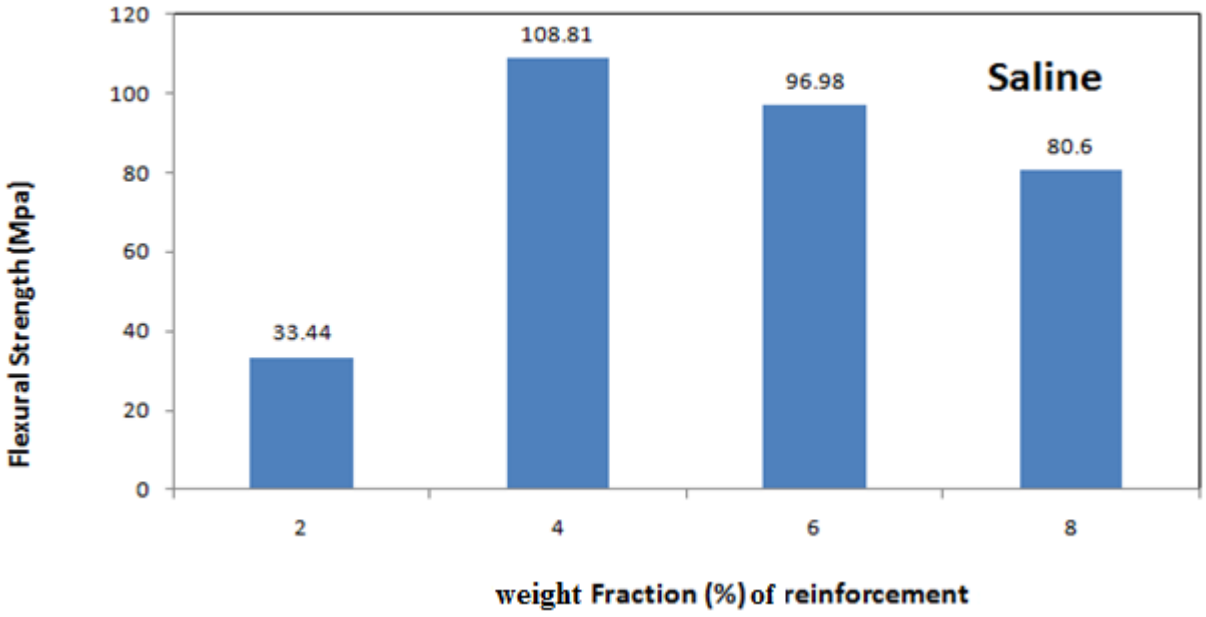


Figure-3.16 Variation of Flexural Strength of different weight fraction in saline environment

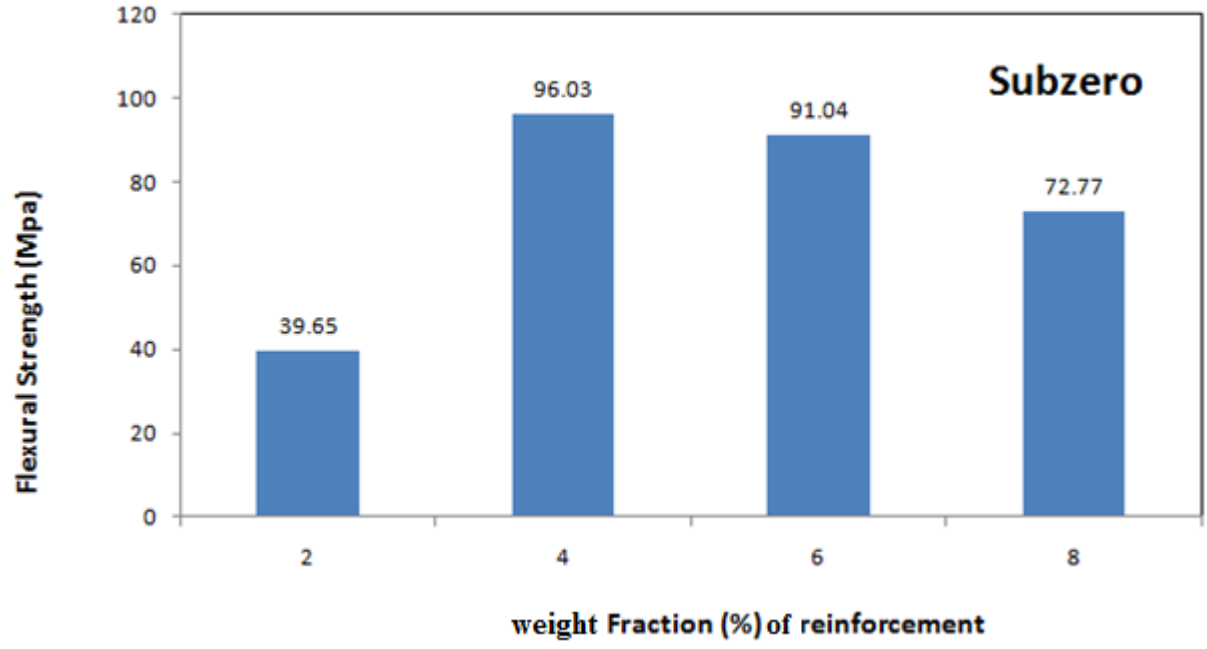


Figure-3.17 Variation of Flexural Strength of different weight fraction in subzero environment

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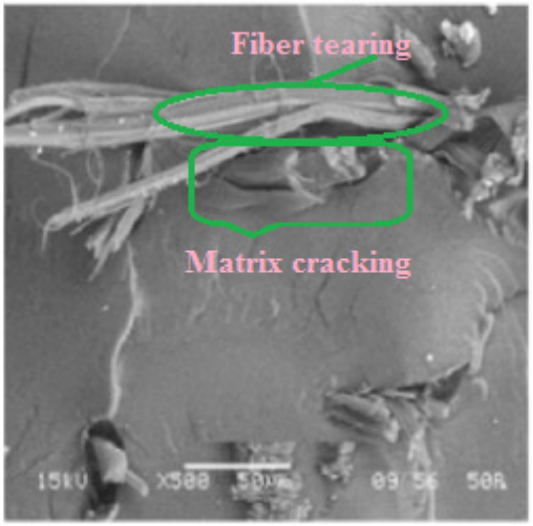


Figure-3.18 (a)

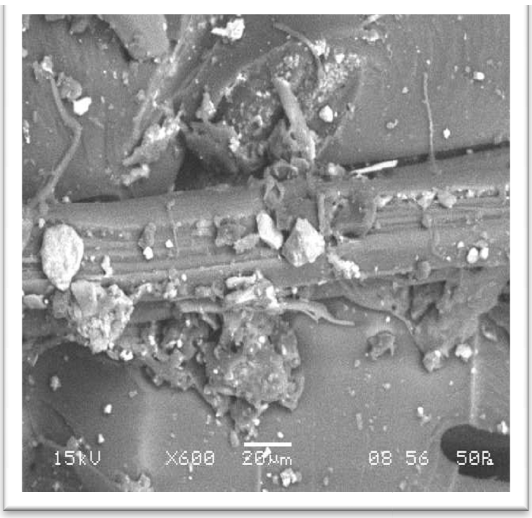


Figure-3.18 (b)

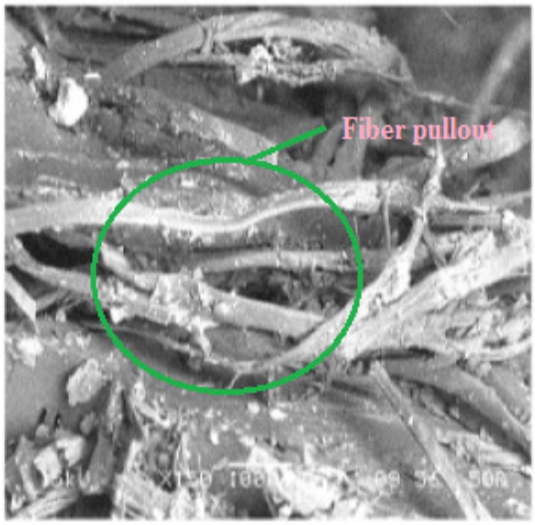


Figure-3.18 (c)

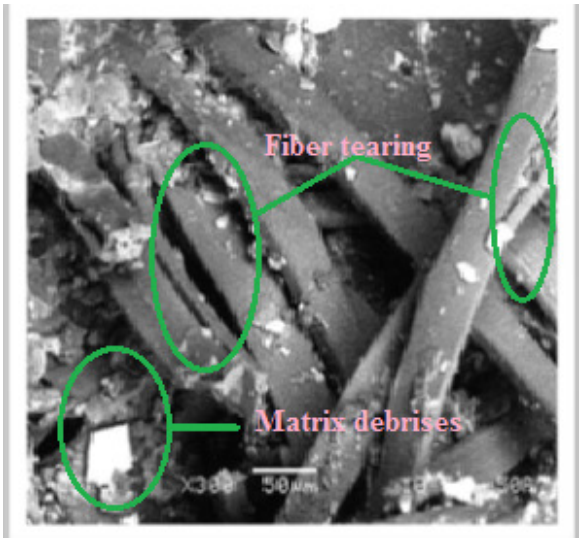


Figure-3.18 (d)

Figure-3.18 Fracture surface of composites under various treatments

CHAPTER – 4

EROSION BEHAVIOUR

4.1 INTRODUCTION

If solid particles impinge against a target surface and causes local damage combined with material removal, this kind of wear is generally termed as erosion. Polymer composites finding wider applications particularly in aerospace and automobile sectors because they provide higher strength and stiffness in comparison to monolithic metals and alloys. These composites are therefore used in different wear situations. Solid particle erosion is one such area. Example of such applications are pipeline carrying sand slurries in petroleum refining, helicopter rotor blades, high speed vehicles , aircrafts operating at desert environments and water turbines [61]. However, polymer composite materials exhibit poor erosion resistance as compared to metallic materials [56]. It is also known that the erosion wear of polymer composite is usually higher than that of the unreinforced polymer matrix [62].

The most important factors influencing the erosion rate of the composite materials can be summarized under four categories; (i) The properties of the target materials (matrix material properties and morphology, reinforcement type, amount and orientation, interface properties between the matrices and reinforcements, etc.), (ii) Environment and testing conditions (temperature, chemical interaction of erodent with the target), (iii) Operating parameters (angle of impingement, impinging velocity, particle flux–mass per unit time, etc.) and (iv) The properties of the erodent (size, shape, type, hardness, etc). [62, 63, 64, 65]

Visualizing the importance of polymeric composites, much work has been done to evaluate various types of polymers and their composites to solid particle erosion [51, 65, 66, 67]. Most of these workers have carried out a wide range of thermoset and thermoplastic PMCs having glass, carbon, graphite and Kevlar fibers in the form of tape, fabric and chopped mat as reinforcement. Also there are limited amount of work done on erosion wear behaviour of natural fiber composite [68, 69]. Hence in the present investigation an attempt has been made to study the erosive wear behavior of waste silk fiber reinforced epoxy composite. In this work it is aimed to study the influence of impinging velocity, impingement angle and fiber loading on erosive wear behavior.

4.2 EXPERIMENT

4.2.1 Preparation of the test specimen

The procedure of making composite is already discussed in section-3.2. From the prepared samples test specimen of size 20x20x5 mm were cut for erosion studies.

4.2.2 Test apparatus and experiment

The schematic figure of the erosion test apparatus used for the present investigation designed as per ASTM-G76 standard. The pictorial of the erosion test rig is shown in Figure-4.1. The test rig consists of an air compressor, a particle feeder, and an air particle mixing and accelerating chamber. The compressed dry air is mixed with the erodent particles, which are fed at a constant rate from a conveyor belt-type feeder in to the mixing chamber and then accelerated by passing the mixture through a tungsten carbide converging nozzle of 4 mm diameter. These accelerated particles impact the specimen, and the specimen could be held at various angles with respect to the impacting particles using an adjustable sample holder. The test apparatus has also been fitted with a rotating double disc to measure the velocity of the erodent particle. The impact velocities of the erodent particles has been evaluated as explained by Ives and Ruff [70] experimentally using this rotating double disc. The velocities obtained from this method for various pressures are given in Table-4.1.

The conditions under which the erosion test is carried out are given in Table-4.2. A standard test procedure is employed for each erosion test. The samples are cleaned in acetone, dried and weighed to an accuracy of 0.001 gm using an electronic balance, prior and after each test. The test samples after loading in the test rig were eroded for 1 min. at a given impingement angle and then weighed again to determine weight loss. The ratio of the weight loss to the weight of the erodent particles causing the material loss is then computed as a dimensionless incremental erosion rate.

This procedure is repeated till the erosion rate attains a constant steady-state value. The erosion efficiency (η) for the process was obtained by using the equation:

$$\eta = \frac{2E_r H}{\rho \times v^2} \tag{4.1}$$

where 'E_r' is erosion rate , 'H' is hardness of eroding material and 'v' is velocity of impact, proposed by Sundararajan et al. [71] Experimental results of the erosion test for different weight fraction of waste silk fiber reinforced epoxy composites with different impingement angle and velocities are tabulated and presented in Table-4.3 to 4.6. These tables also show the average values of the erosion rate.

Table–4.1

Impact velocity calibration at various pressures

Pressure (bar)	**Speed of rotating disc(rpm)**	**Angle θ (°)**	**Velocity(m/s)**	**Avg. impact velocity(m/s)**
1 bar	2000	7.0	42.85	47.25
		6.5	46.15	
		6.0	50.00	
		6.0	50.00	
2 bar	2000	4.0	75.00	69.16
		4.5	66.67	
		4.0	75.00	
		5.0	60.00	
3 bar	2000	4.5	66.67	81.845
		4.0	75.00	
		3.5	85.71	
		3.0	100.00	

Table-4.2

Test parameters

Erodent	Silica sand
Erodent size (µm)	200 ± 50
Impingement angle (α^0)	30, 45, 60, 90
Impact velocity (m/s)	48, 70, 82
Erodent feed rate (g/min)	4
Test temperature	Room temperature
Nozzle to sample distance (mm)	10
Nozzle diameter(mm)	4
Time	5min.

1. Sand hopper

2. Conveyor belt system for sand flow

3. Pressure transducer

4. Particle-air mixing chamber

5. Nozzle

6. X–Y and h axes assembly.

7. Sample holder

Figure-4.1 Solid Particle Erosion Test Set up

Table-4.3

Erosion rate of the samples at 30^0 impingement angle

Velocity (m/s)	Cumulative Weight (g)	Erosion Rate x 10^{-4} (g/g)				
		Epoxy	**2%**	**4%**	**6%**	**8%**
48	40.50	4.25	3.95	2.96	2.47	1.48
	81.00	4.65	4.12	3.09	2.47	1.65
	121.50	4.89	4.32	3.29	2.96	1.85
	162.00	4.99	4.94	3.7	3.09	2.47
	202.50	5.12	4.94	4.94	3.29	2.47
Average		4.78	4.454	3.596	2.856	1.984
70	40.5	4.1	3.7	2.96	2.47	1.65
	81.00	4.22	4.12	3.09	2.47	1.85
	121.5	4.46	4.32	3.29	3.09	2.47
	162.00	4.89	4.44	3.7	3.29	2.47
	202.50	5.22	4.94	4.44	3.46	2.47
Average		4.974	4.304	3.496	2.956	2.182
82	40.5	4.2	3.7	3.7	2.47	3.29
	81.00	4.35	4.12	3.7	3.46	3.7
	121.5	4.55	4.32	3.95	3.7	3.7
	162.00	4.78	4.44	4.12	3.7	3.95
	202.50	5.33	4.94	4.94	4.12	4.94
Average		4.642	4.304	4.082	3.49	3.916

Table-4.4

Erosion rate of the samples at 45^0 impingement angle

Velocity (m/s)	Cumulative Weight (g)	Erosion Rate x 10^{-4} (g/g)				
		Epoxy	**2%**	**4%**	**6%**	**8%**
48	40.50	5.33	4.94	3.95	3.7	2.47
	81.00	5.45	4.94	4.12	4.12	3.7
	121.50	5.66	4.94	4.32	4.32	4.12
	162.00	5.99	5.43	4.94	4.44	4.32
	202.50	6.1	5.56	4.94	4.94	4.44
Average		5.706	5.162	4.454	4.304	3.81
70	40.5	6.89	6.58	6.17	4.94	4.94
	81.00	7.1	6.79	6.17	4.94	4.94
	121.5	7.89	7.41	6.58	4.94	4.94
	162.00	8.1	7.41	6.91	4.94	5.76
	202.50	8.3	7.41	7.41	5.43	6.17
Average		7.656	7.12	6.648	5.038	5.35
82	40.5	8.9	8.4	8.32	4.94	4.94
	81.00	9.5	9.26	8.4	4.94	4.94
	121.5	10.9	10.7	8.64	4.94	5.56
	162.00	12.1	11.11	8.64	4.94	5.76
	202.50	15.2	14.81	9.88	5.43	6.17
Average		11.32	10.856	8.776	5.038	5.474

Table-4.5

Erosion rate of the samples at 60^0 impingement angle

Velocity (m/s)	Cumulative Weight (g)	Erosion Rate x 10^{-4} (g/g)				
		Epoxy	**2%**	**4%**	**6%**	**8%**
48	40.50	4.2	3.7	3.46	2.47	1.98
	81.00	4.6	4.12	3.7	2.47	2.47
	121.50	4.9	4.32	3.7	2.47	2.47
	162.00	4.98	4.44	4.12	2.96	2.47
	202.50	5.2	4.94	4.94	3.09	2.47
Average		4.776	4.304	3.984	2.692	2.372
70	40.5	5.6	4.94	3.7	3.7	2.47
	81.00	5.8	4.94	4.12	3.7	3.46
	121.5	6.2	5.43	4.32	3.95	3.7
	162.00	6.9	5.76	4.94	4.12	3.7
	202.50	7.2	6.17	4.94	4.94	4.12
Average		6.34	5.448	4.404	4.082	3.49
82	40.5	7.9	6.58	3.7	2.92	3.29
	81.00	7.98	6.79	4.12	2.95	3.7
	121.5	8.2	7.41	4.32	2.97	3.7
	162.00	8.3	7.41	4.44	3	3.95
	202.50	8.5	7.41	4.94	3.03	4.94
Average		8.176	7.12	4.304	2.974	3.916

Table-4.6

Erosion rate of the samples at 90^0 impingement angle

Velocity (m/s)	Cumulative Weight (g)	Erosion Rate x 10^{-4} (g/g)				
		Epoxy	**2%**	**4%**	**6%**	**8%**
48	40.50	4.5	3.46	2.47	2.47	2.47
	81.00	4.8	3.7	3.29	2.47	2.47
	121.50	4.9	3.7	3.46	2.96	2.47
	162.00	5.2	4.12	3.7	3.09	2.47
	202.50	5.9	4.94	3.7	3.29	3.29
Average		5.06	3.984	3.324	2.856	2.634
70	40.5	6.2	4.94	3.46	2.47	2.47
	81.00	6.3	4.94	3.7	2.47	2.7
	121.5	6.5	4.94	4.12	2.47	3.12
	162.00	6.9	5.43	4.94	2.96	3.32
	202.50	7.2	6.17	4.94	3.09	3.44
Average		6.62	5.284	4.232	2.692	3.01
82	40.5	5.4	4.94	4.94	2.47	3.7
	81.00	6.8	5.93	4.94	2.47	3.7
	121.5	7.2	6.17	5.43	3.09	3.95
	162.00	7.6	6.17	5.76	3.29	4.12
	202.50	7.9	6.58	6.17	3.46	4.94
Average		6.98	5.958	5.448	2.956	4.082

Table-4.7

Erosion efficiency (η) of samples

Impingement Angle	Velocity (m/s)	Erosion Efficiency (η) in %				
		Epoxy	2%	4%	6%	8%
30^0	48	6.3456	1.843687	2.0169	1.3376	1.94478
	70	14.5464	2.833331	2.58827	1.606779	2.94478
	82	16.124	4.495519	4.0426	2.3758	4.50769
45^0	48	6.985	4.5785	3.7593	2.9604	2.696
	70	15.764	5.0872	3.90785	3.73887	3.929
	82	20.623	6.09127	6.4532	6.792	5.951
60^0	48	8.231	3.503	2.1178	1.2739	2.303
	70	12.9866	4.5785	3.2675	2.3995	2.564
	82	19.864	5.4485	4.247	4.381	3.7428
90^0	48	9.142	2.792	2.334	1.977	1.598
	70	11.965	3.3978	2.4877	2.1846	2.826
	82	16.983	5.448	4.1555	4.114	4.5067

4.3 RESULT AND DISCUSSION

Based on the tabulated results various graphs were plotted and presented in Figure-4.2 to 4.24 for different percentage of reinforcement under different test conditions.

Figure- 4.2- 4.13 shows the variation of erosion rate of all the composite (i.e. 2,4,6 and 8wt%) as a function of the cumulative weight of impinging particles with different impingement angle(30^0, 45^0,60^0 and 90^0) with different impact velocities (i.e. 48, 70 and 82m/s). The plots were obtained by determining the steady state of the weight loss. The nature of the curve is also different for different materials tested .In all these curves no incubation or induction period is observed for different material tested. Instead figures clearly show that the response of material to the weight of the erodent was acceleration and stabilization. It also shows higher erosion rate with increase in impact velocity for different target materials.

The variation of steady-state erosion rate of both neat epoxy and silk fiber reinforced epoxy as a function of angle of impingement under different impact velocities are shown in Figure-4.14 to 4.17. It is evident from the plot that erosion rate of all composite samples as well as for pure epoxy increases with increase in the impact velocity. However, neat epoxy shows least variation in the erosion rate with increase in the impact velocity at low impact angle ($\alpha = 30^0$). Also, it is clear from the plot that the best erosion resistance under all impact conditions is achieved for the composite made of 8wt% reinforced silk fiber. Irrespective of impingement angle and impact velocity, there is a steady decrease in erosion rate with increase in fiber content has also been observed. This indicates that erosion rate of composite is dominated only by the weight fraction of fiber (silk). Similar type of observation was reported by Miyazaki et al. [72] while worked with glass and carbon fiber reinforced polyetheretherketon composites. It is also interesting to note that for the 6 wt% reinforced fibers the erosion rate is found to be less at velocity of 82 m/s for all the impingement angles.

Figure-4.18 to 4.20 illustrate the erosion wear rates of both neat epoxy and silk reinforced epoxy composite as a function of impingement angle under different impact velocities (48m/s to 82m/s). It is observed that silk fiber epoxy composite shows peak erosion rate (E_r max) at 45^0 impact angle and minimum erosion rate (E_r min) at 30^0 under all velocity of impact. Generally, it has been recognized that peak erosion exists at low impact angles (15^0–30^0) for ductile materials

and at a high impact angle (90^0) for brittle materials [73]. However the maximum erosion occurring in the angular range 45^0– 60^0, it describes the semi-ductile behaviour of the material [53]. From the experimental results it is clear that silk fiber reinforced composites respond to solid particle impact in a semi brittle manner since the maximum erosion occurs at 45° impact angle for all the velocity range. However the erosion rate is found to be different for different velocities. The same type of behavior was also reported by Pool et al. [74] while studying the UD and woven graphite reinforced epoxy composite. Deo and Acharya [68] while studying the erosive wear behavior of Lantana camara fiber reinforced epoxy composite showed that their composite behaves in a semi ductile in nature. Thus it can be concluded that the behavior of natural fiber composite to solid particle erosion depends on type of fiber. It is further noticed that irrespective of impact velocity and impact angle, the erosion rate is lowest for 8 % silk fiber reinforced epoxy composite.

It has been reported by Sundararajan et al [71] that the erosion efficiency (η) can be used to characterize the nature and mechanism of erosion. They also showed that the ductile material possess a very low erosion efficiency (ie) $\eta <<< 100\%$. The values of erosion efficiencies of composites under this study are calculated using equation 4.2 and are listed in table-4.7 along with their hardness values and operating conditions. The variation of erosion efficiency of all composite samples with impact velocity at different impact angles are shown in the form of a histogram in Figure-4.21 to 4.24. It is observed that the erosion efficiency increases with increasing the impact velocities. It has also been observed that the erosion efficiencies of silk reinforced composites vary from 1.2739 to 6.792 % for different impact velocities studied at different angles. Similar observations are also reported by Srivastava et al. [54] for glass fiber reinforced fly-ash filled epoxy composite. Thus it can be conclude that the erosion efficiency is not exclusively a material property; but also depends on other operational variables such as impact velocity and impingement angle. The data shown in Table-4.7 are also indicates that the erosion efficiency of silk fiber epoxy composite decreases with increase in fiber content up to 6wt% silk fiber and increases again for 8wt% fiber whereas the neat epoxy exhibits a higher value under all testing condition. The lower erosion efficiency of silk fiber at 6 wt% reinforcement can be taken as optimum so far as erosion resistance is concerned. This also supplements to the results shown in Figure-4.14 to 4.17.

To characterize the morphology of eroded surfaces eroded samples were observed under scanning electron microscope. Figure-4.25(a) shows the crater formed and the damage caused to the composite at lower impact angle. Figure-4.25(b) shows cracks that are formed at higher impact angle but detachment of fiber from the matrix is not visible .This may be due to good wet-ability of fiber with the matrix material.

4.4 CONCLUSION

Based on the study of the erosive wear behavior of SFRP composites at various impingement angles, impact velocities for different fiber weight fraction with silica sand as erodent the following conclusions are drawn.

- The composite exhibited a maximum erosion rate at an impingement angle of 45° under present experimental condition indicating semi ductile behavior.

- Fiber weight fraction and velocity of impact has a significant influence on the erosion rate of the composite.

- The erosion efficiency of Silk fiber reinforced epoxy composite decreases with increase in fiber content. The 6% weight fraction of silk fiber epoxy composite indicates a better erosion resistance.

- The morphologies of the eroded surfaces observed by SEM suggest that overall erosion damage of the composite is mainly due to micro cracking.

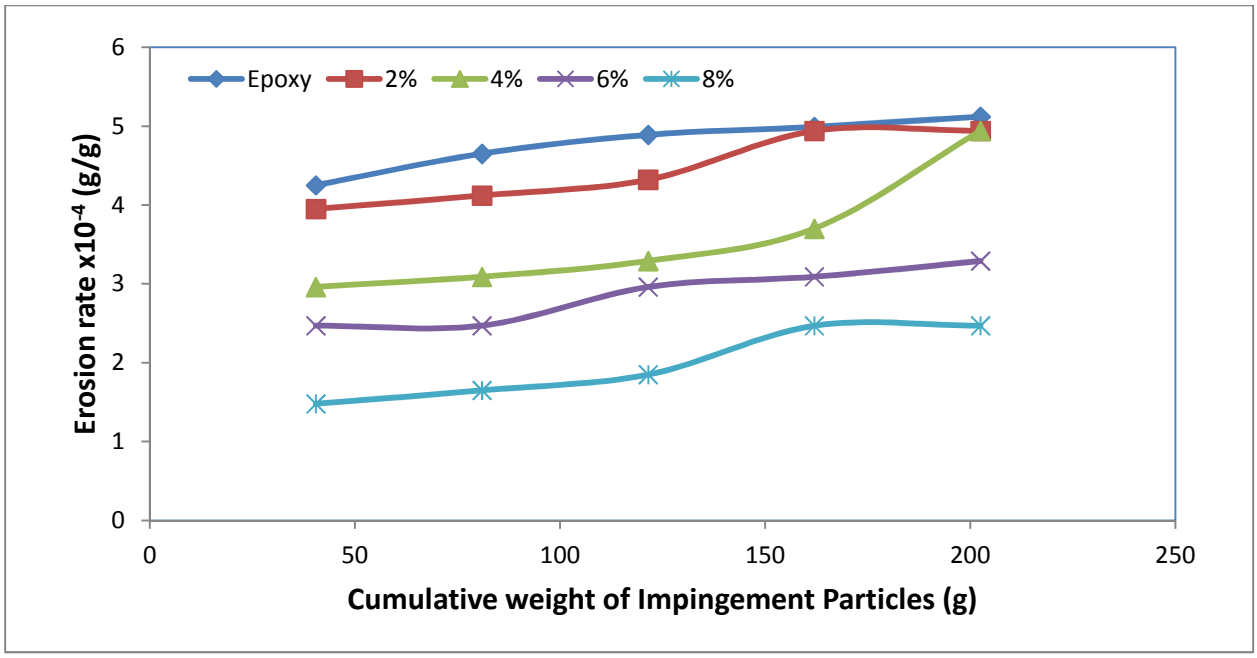


Figure-4.2 Variation of erosion rate with cumulative weight of impingement particle at impingement angle 30^0 at velocity 48m/s

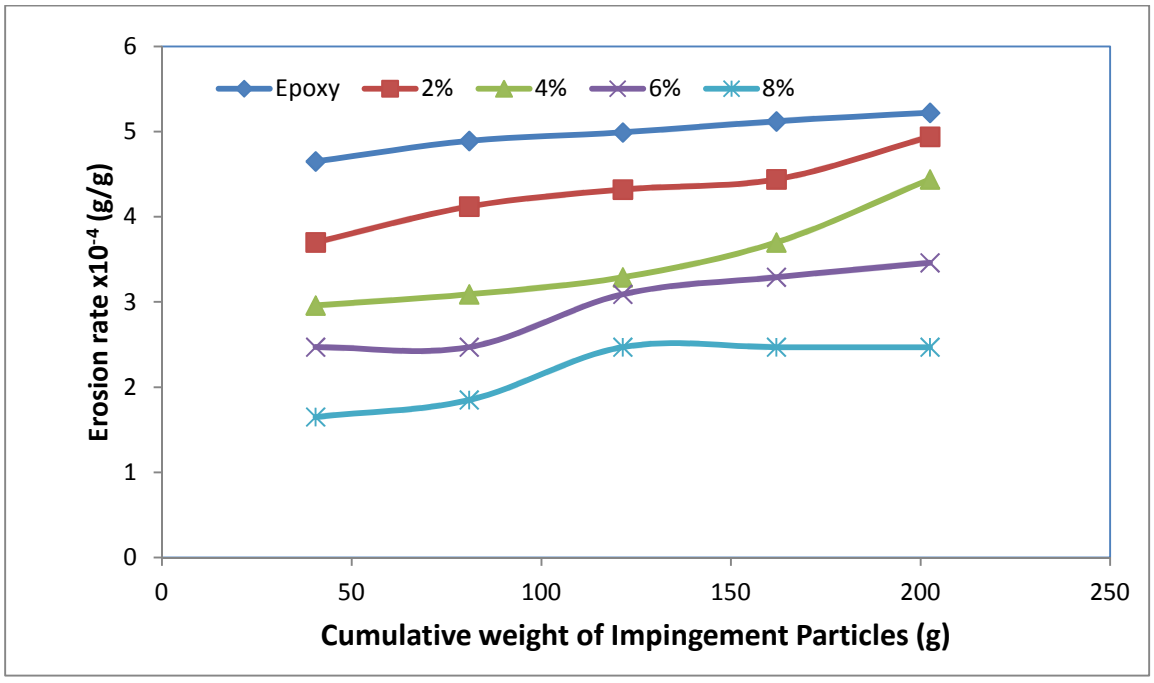


Figure-4.3 Variation of erosion rate with cumulative weight of impingement particle at impingement angle 30^0 at velocity 70m/s

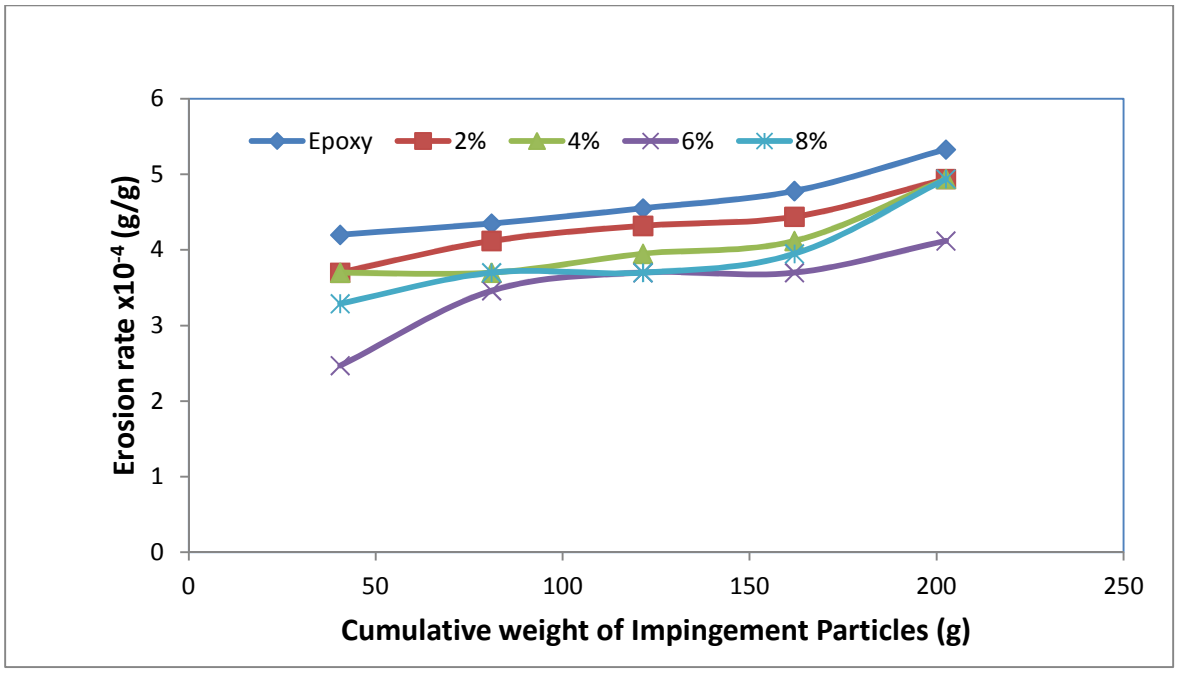


Figure-4.4 Variation of erosion rate with cumulative weight of impingement particle at impingement angle 30^0 at velocity 82m/s

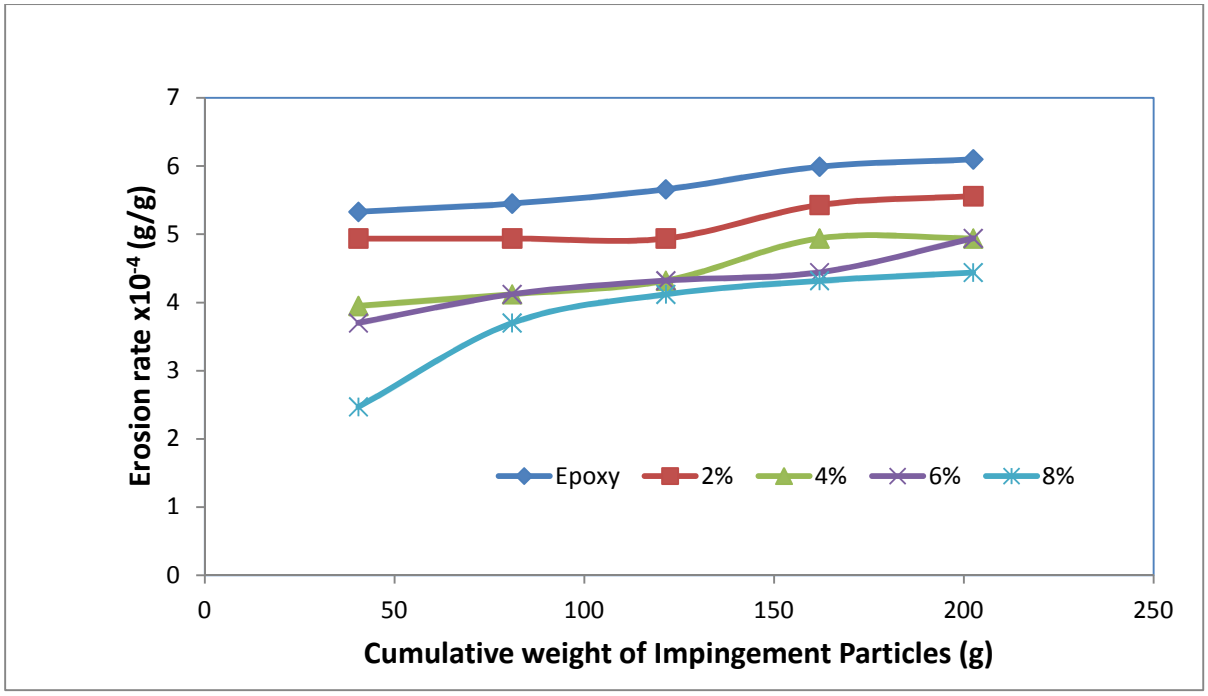


Figure-4.5 Variation of erosion rate with cumulative weight of impingement particle at impingement angle 45^0 at velocity 48m/s.

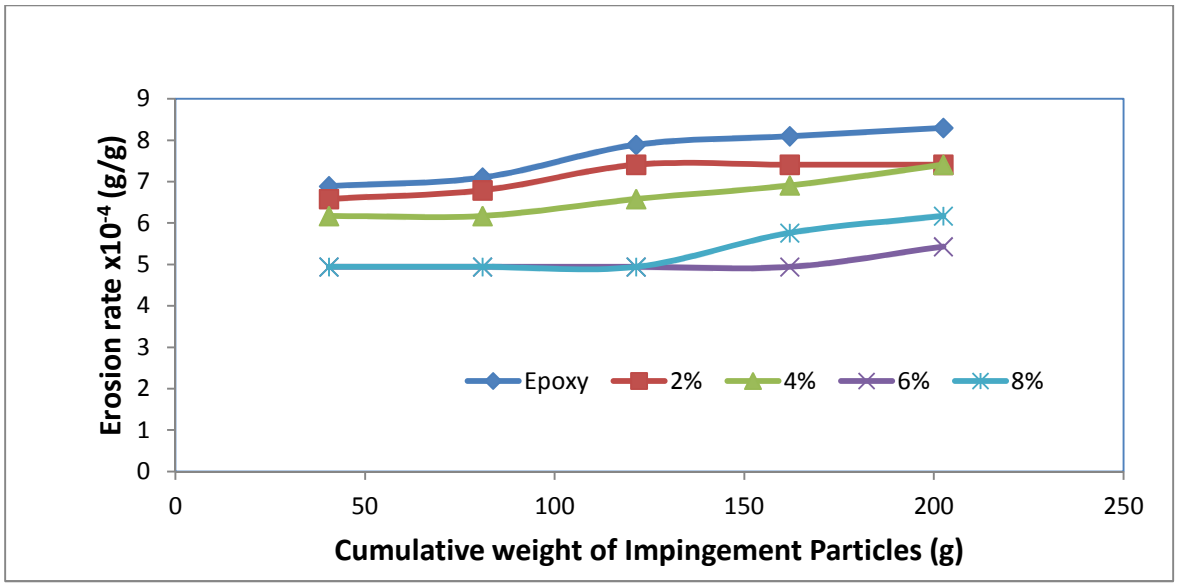


Figure-4.6 Variation of erosion rate with cumulative weight of impingement particle at impingement angle 45^0 at velocity 70m/s

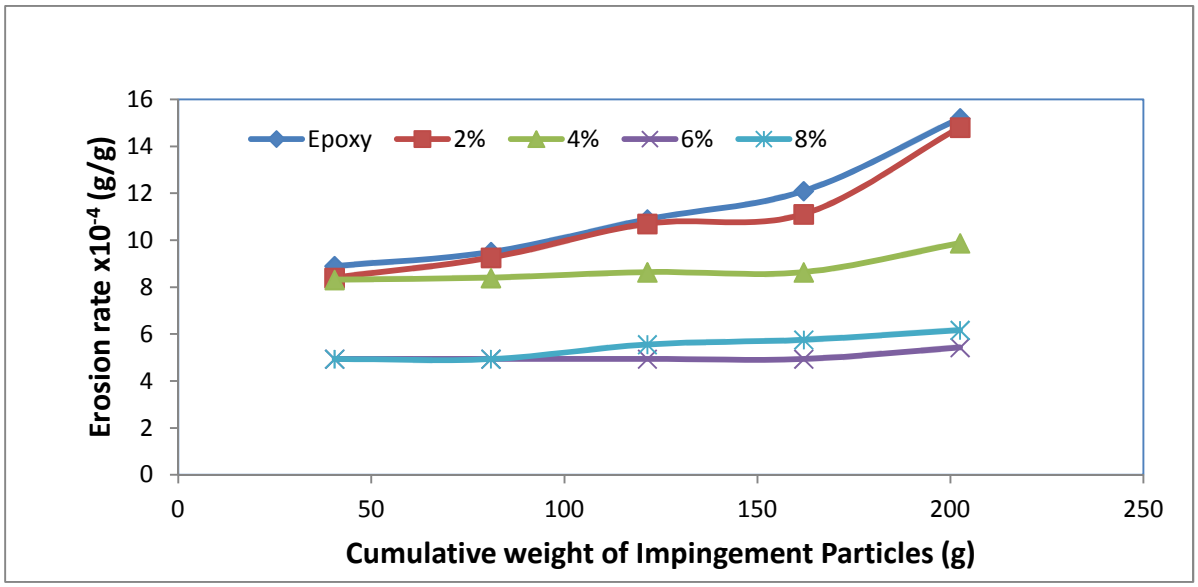


Figure-4.7 Variation of erosion rate with cumulative weight of impingement particle at impingement angle 45^0 at velocity 82m/s

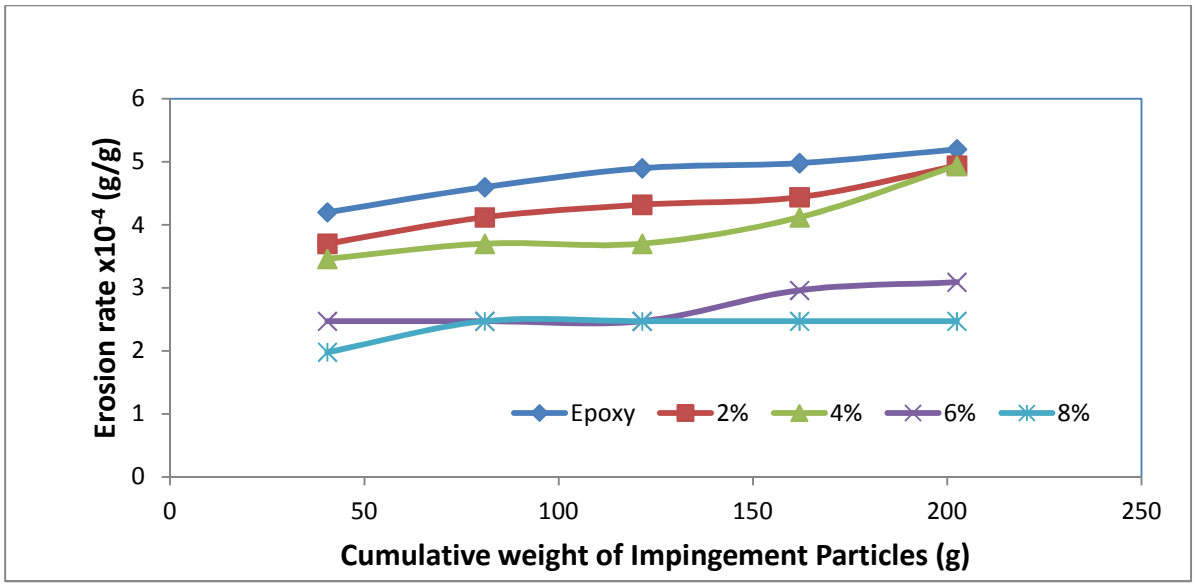


Figure-4.8 Variation of erosion rate with cumulative weight of impingement particle at impingement angle 60^0 at velocity 48m/s.

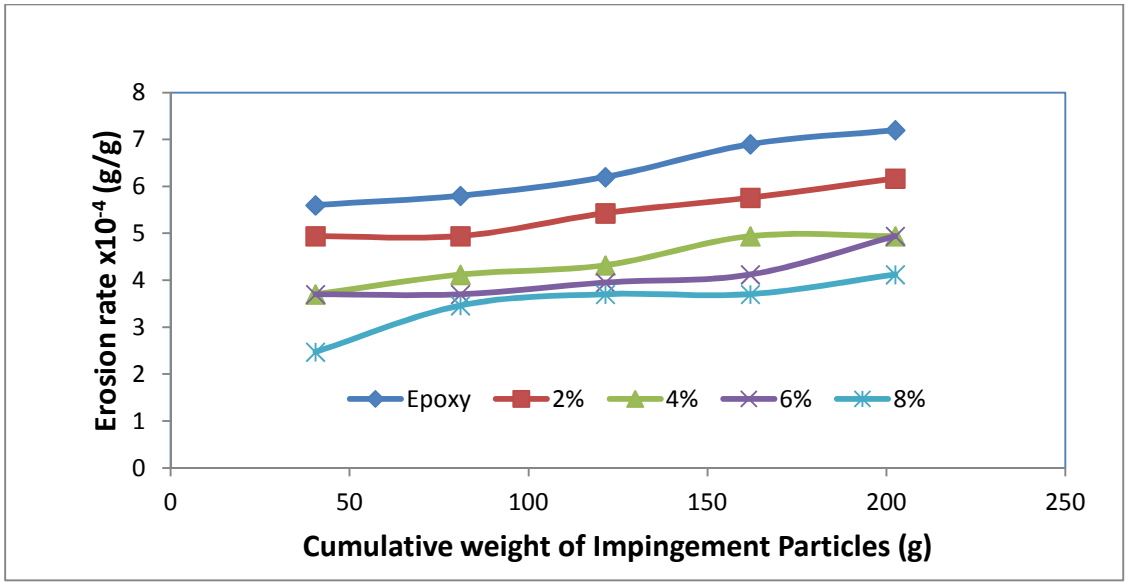


Figure-4.9 Variation of erosion rate with cumulative weight of impingement particle at impingement angle 60^0 at velocity 70m/s.

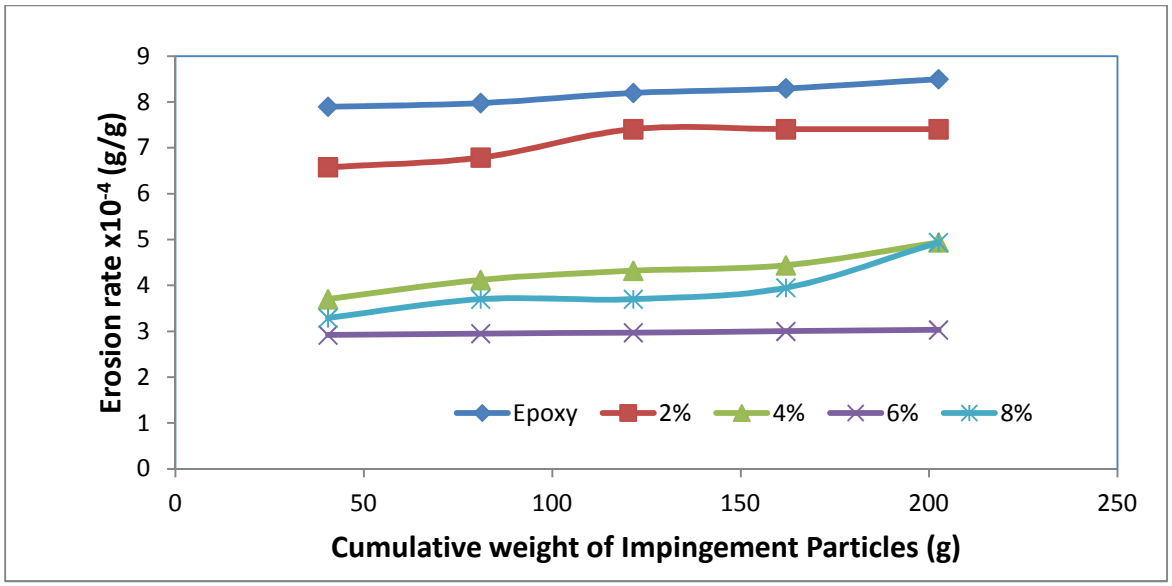


Figure-4.10 Variation of erosion rate with cumulative weight of impingement particle at impingement angle 60^0 at velocity 82m/s.

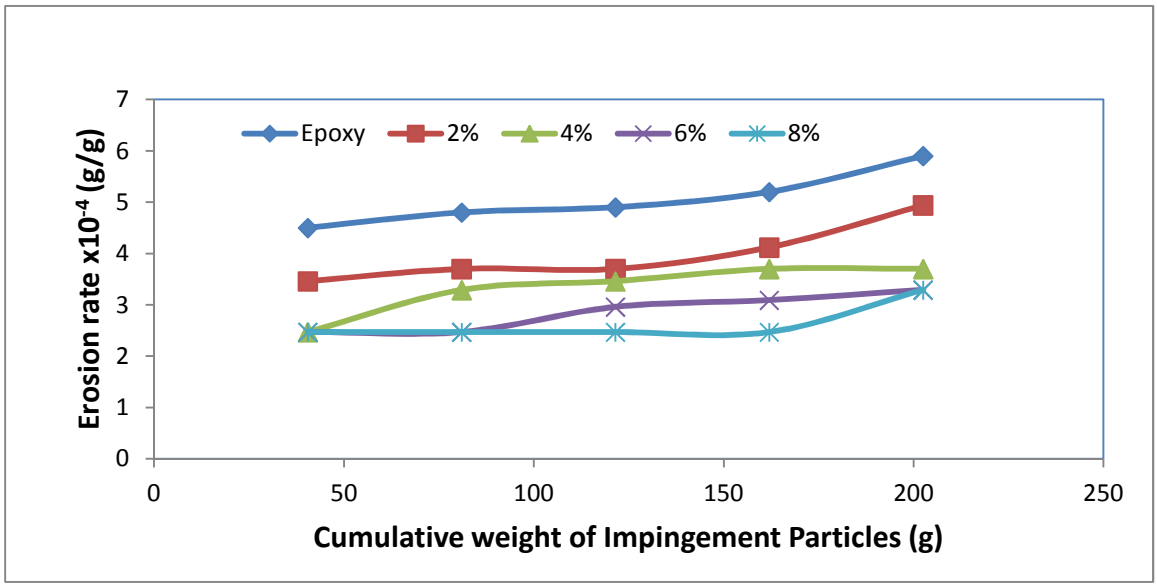


Figure-4.11 Variation of erosion rate with cumulative weight of impingement particle at impingement angle 90^0 at velocity 48m/s.

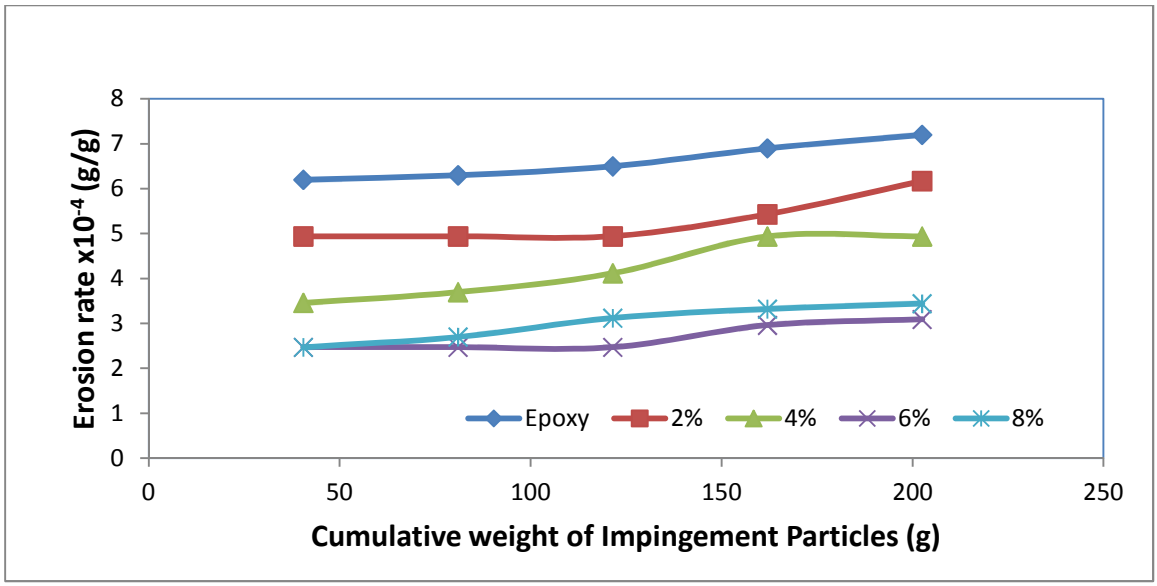


Figure-4.12 Variation of erosion rate with cumulative weight of impingement particle at impingement angle 90^0 at velocity 70m/s

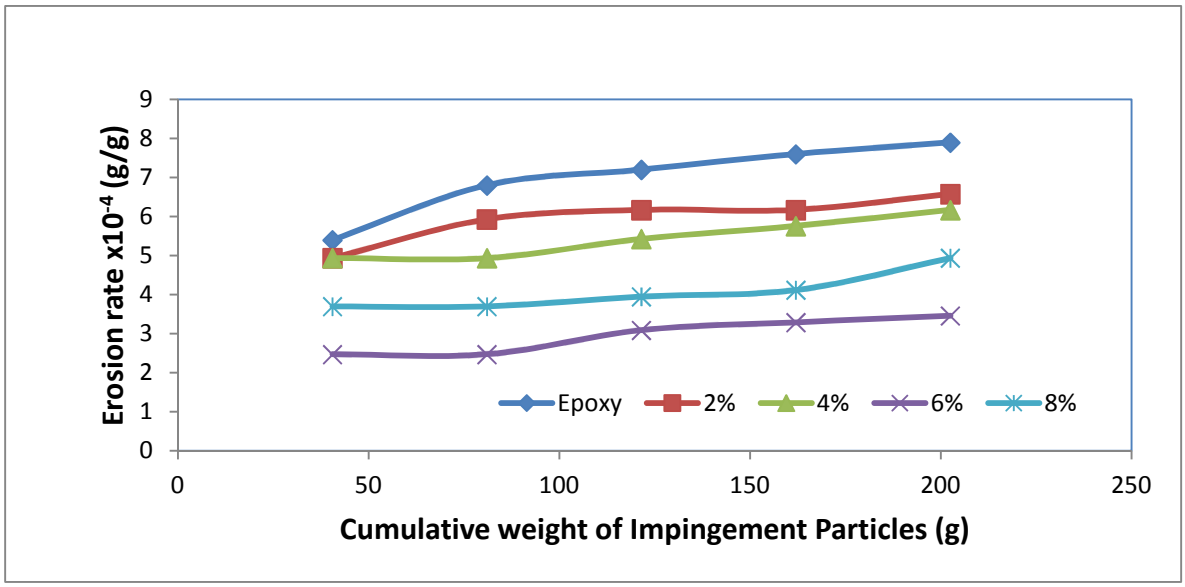


Figure-4.13 Variation of erosion rate with cumulative weight of impingement particle at impingement angle 90^0 at velocity 82m/s

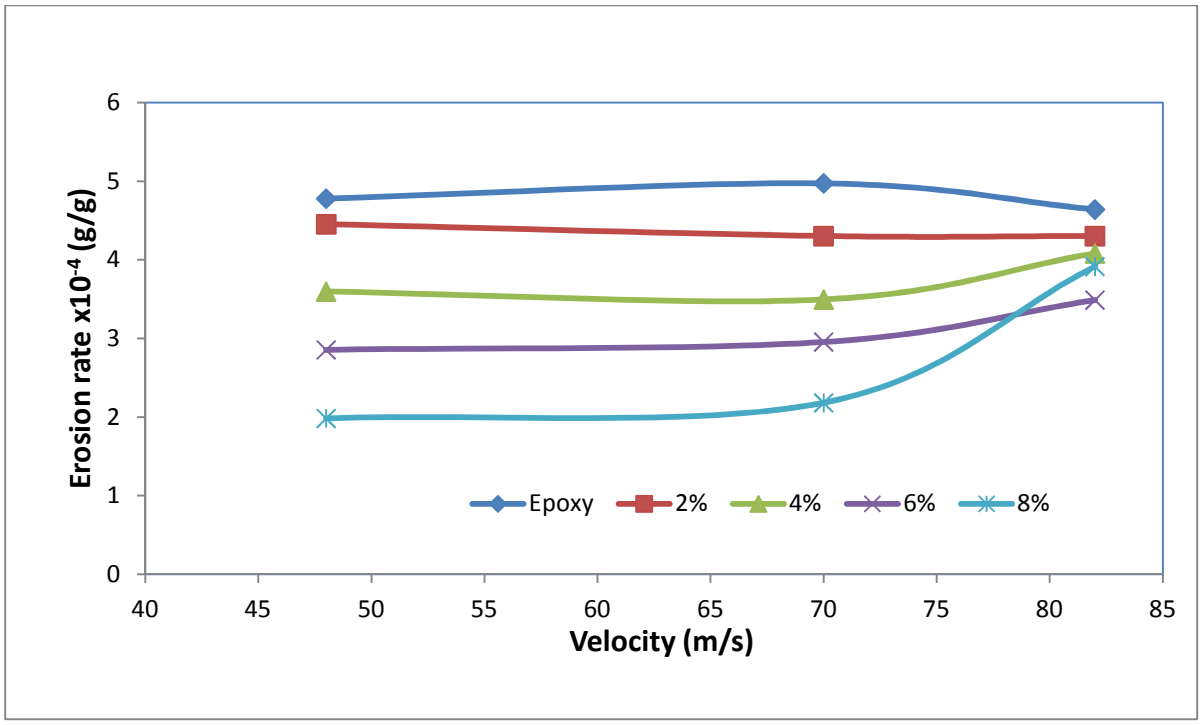


Figure-4.14 Variation of erosion rate with velocity of particle at impingement angle 30^0

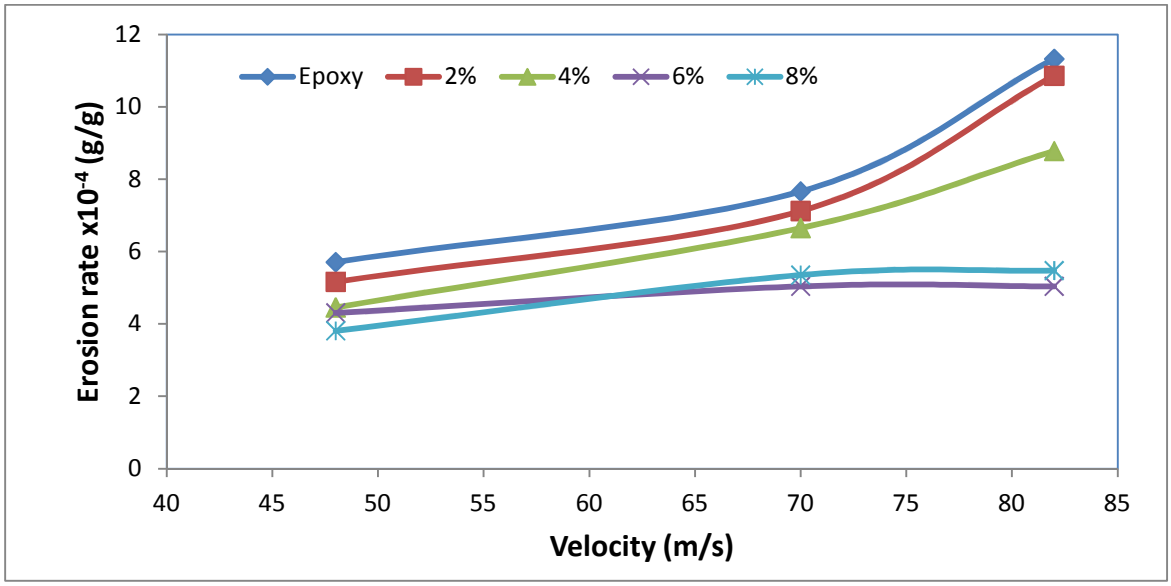


Figure-4.15 Variation of erosion rate with velocity of particle at impingement angle 45^0

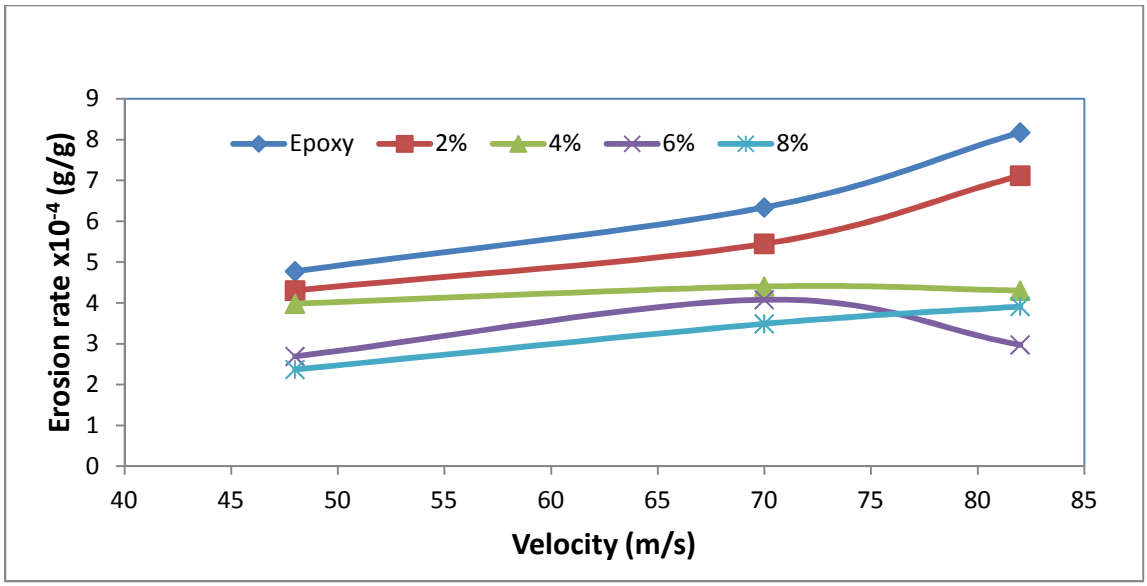


Figure-4.16 Variation of erosion rate with velocity of particle at impingement angle 60^0

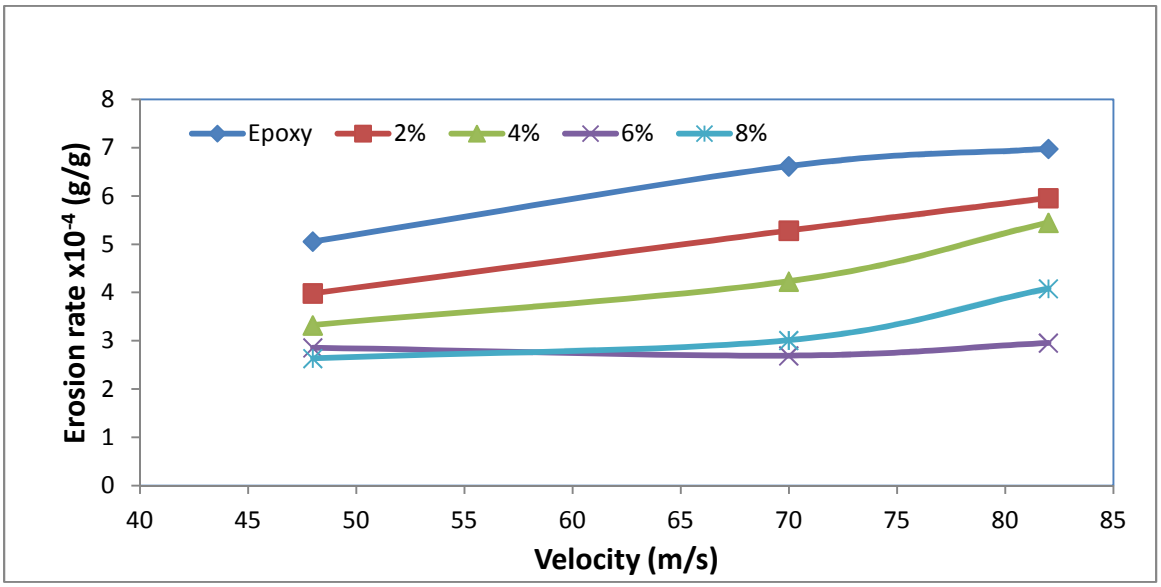


Figure-4.17 Variation of erosion rate with velocity of particle at impingement angle 90^0

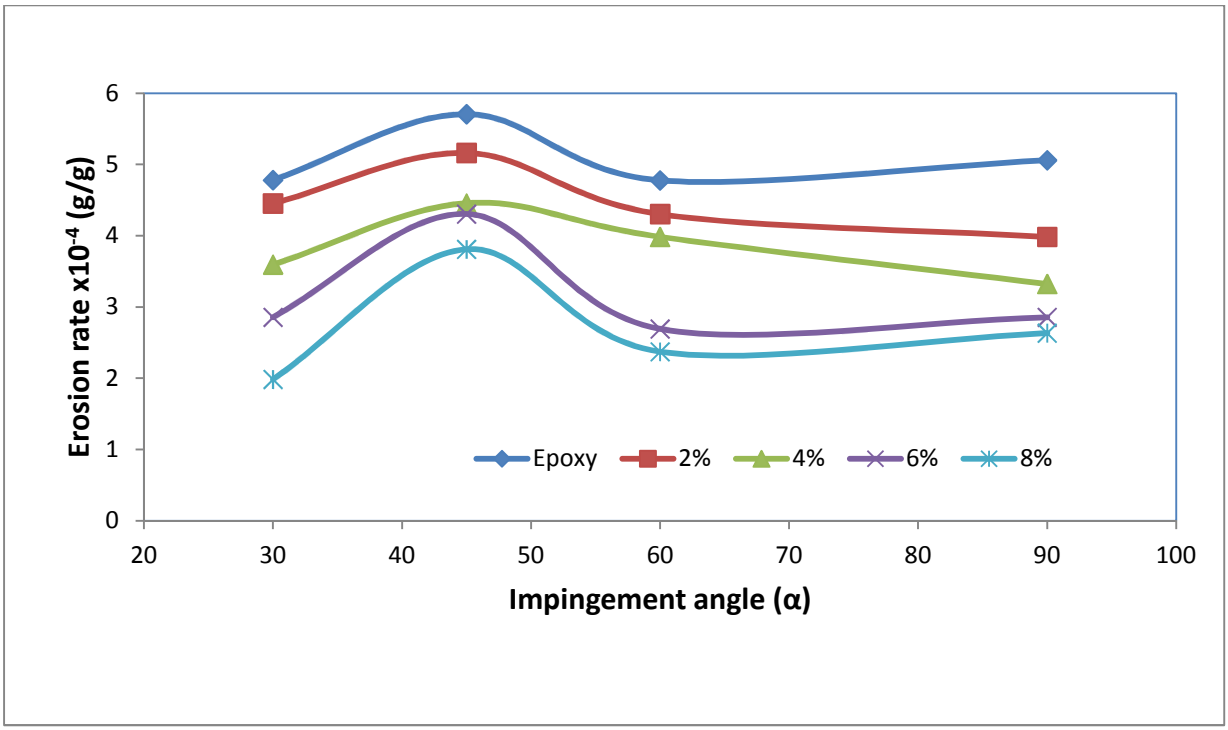


Figure-4.18 Variation of erosion rate with impingement angle at velocity 48m/s

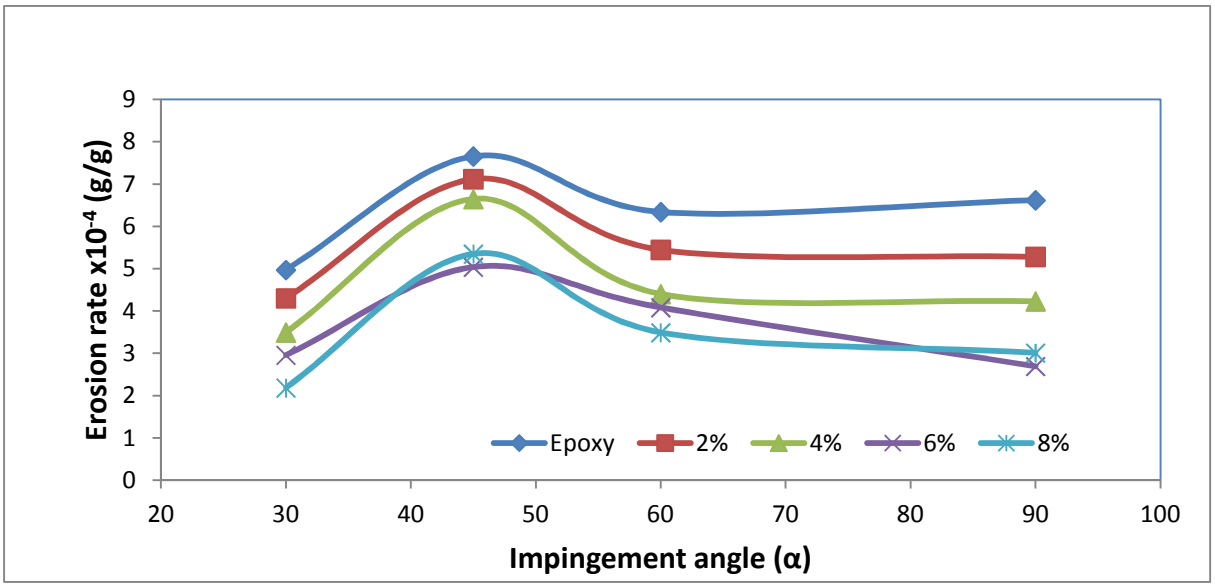


Figure-4.19 Variation of erosion rate with impingement angle at velocity 70m/s

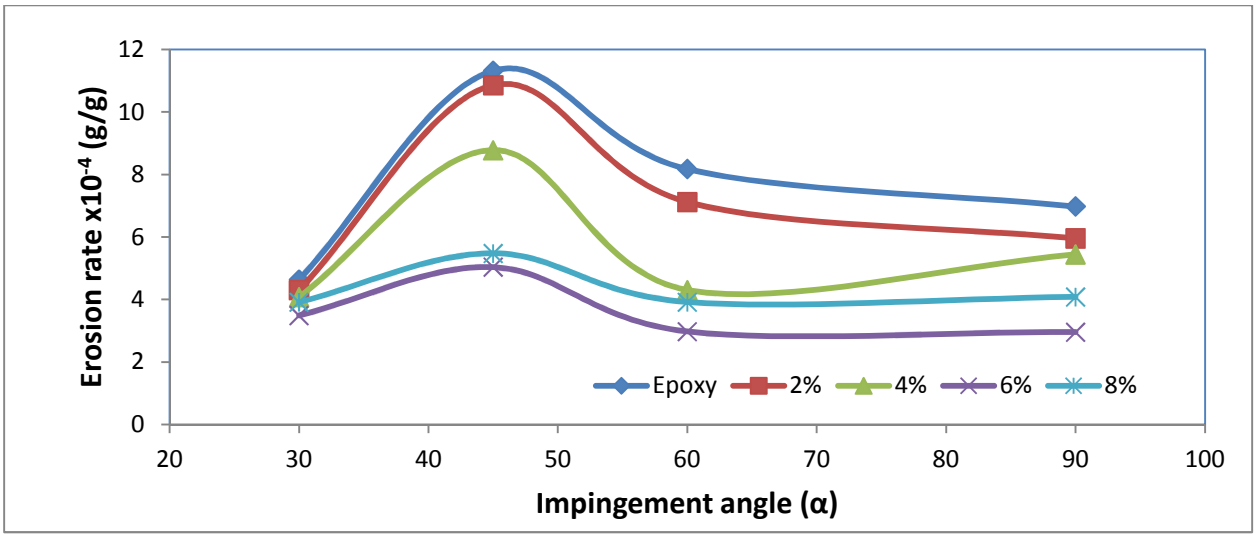


Figure-4.20 Variation of erosion rate with impingement angle at velocity 82m/s

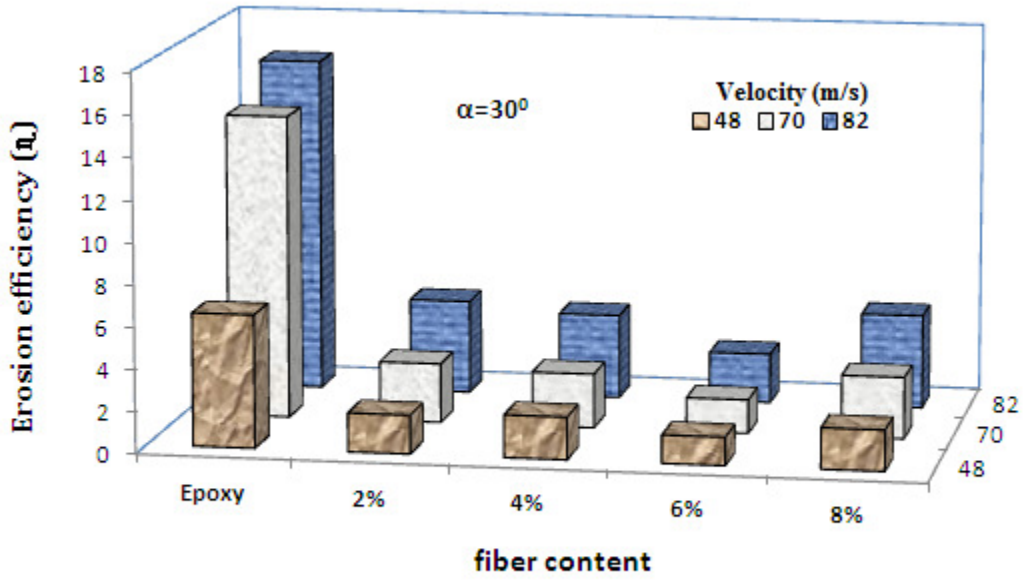


Figure-4.21 Effect of velocity on erosion efficiency with impingement angle 30^0

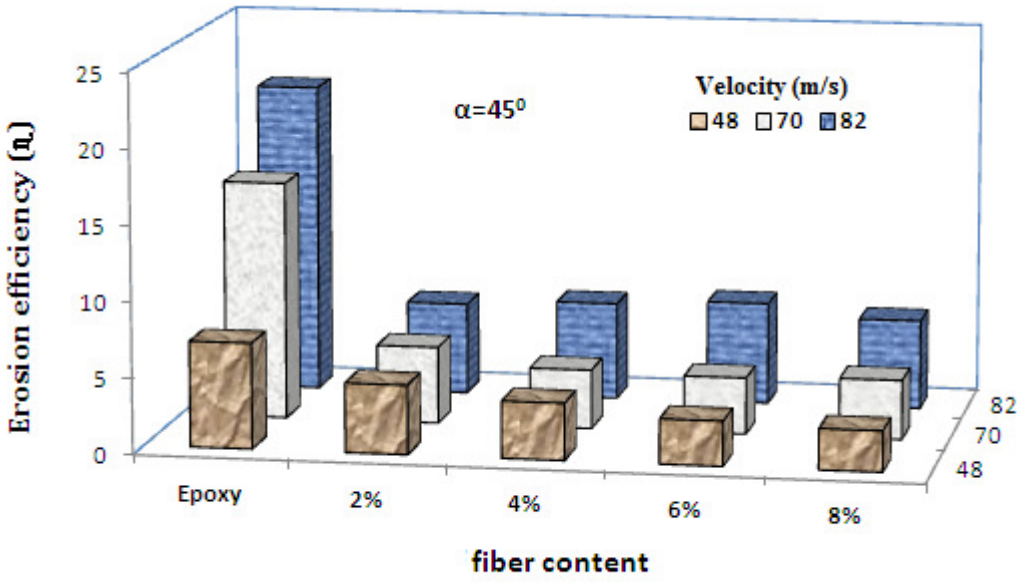


Figure-4.22 Effect of velocity on erosion efficiency with impingement angle 45^0

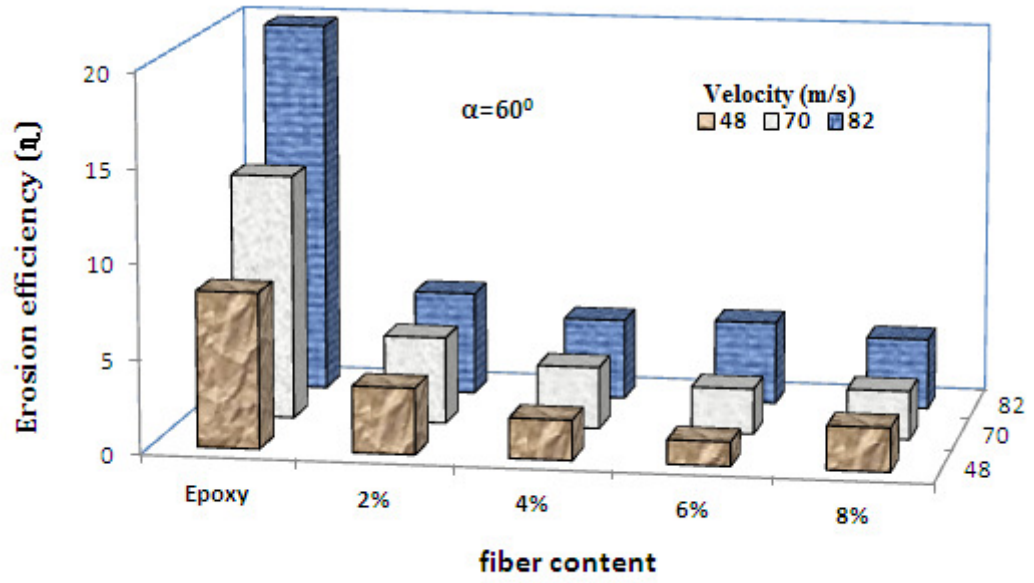


Figure-4.23 Effect of velocity on erosion efficiency with impingement angle 60^0

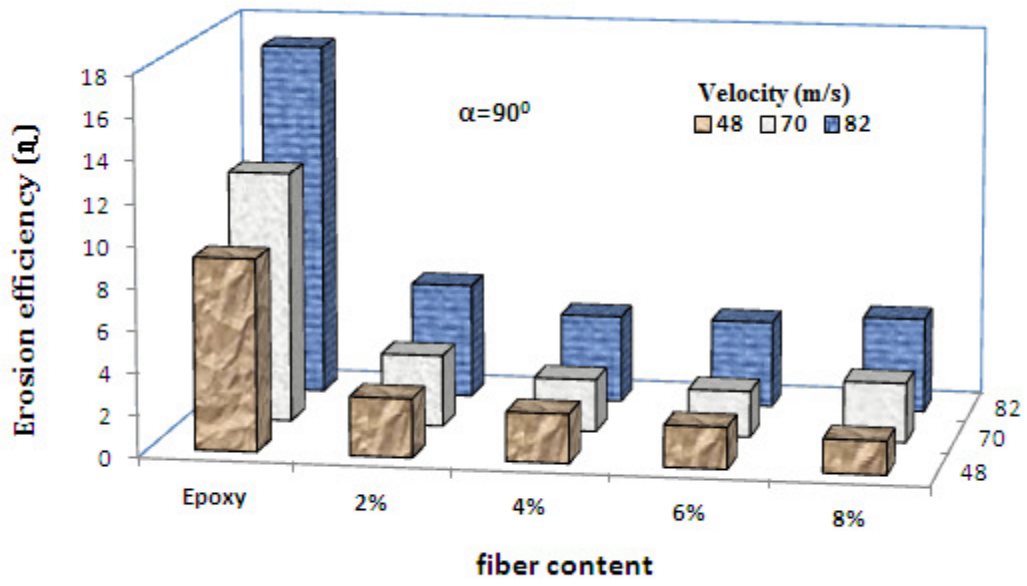
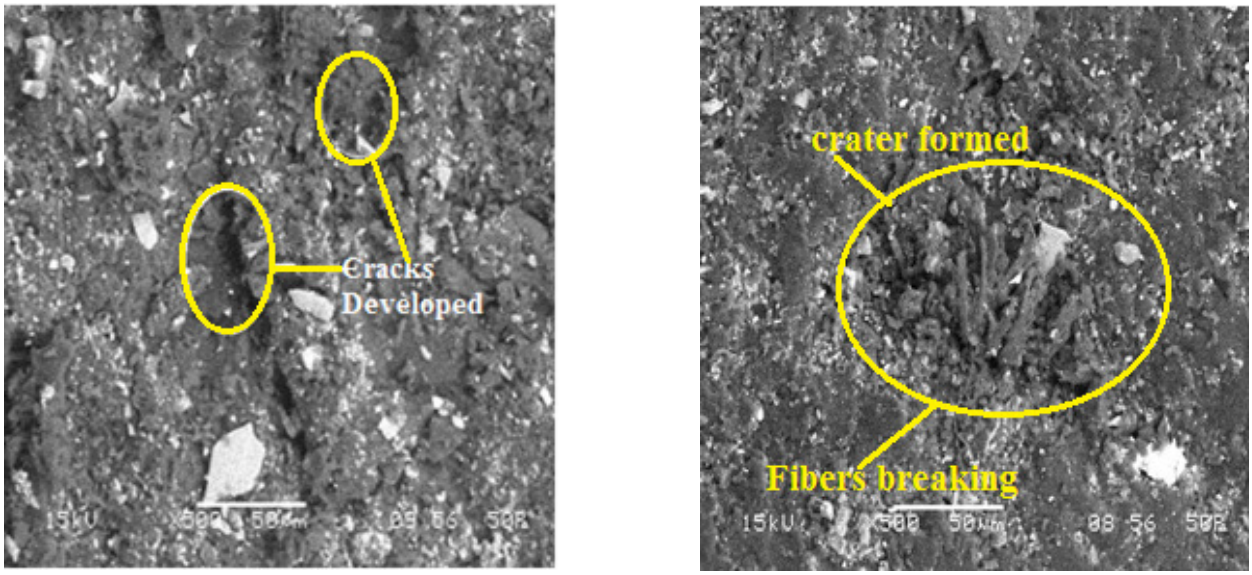


Figure-4.24 Effect of velocity on erosion efficiency with impingement angle 90^0



(a) **(b)**

Figure- 4.25 (a) SEM micrograph of 6wt % fiber surface for 45^0 impact angle.

(b) SEM Micrograph of 6wt % fiber surface for 60^0 impact angle.

CHAPTER – 5

HYBRIDIZATION OF COMPOSITE USING SYNTHETIC AND NATURAL FIBER

5.1 INTRODUCTION:

Natural fibers exhibit many advantageous properties as reinforcement for composites. They are known as low density materials yielding relatively light weight composite with high specific properties [75, 76]. Natural fibers also offer significant cost advantages and benefits associated with processing, as compared to the synthetic fibers such as glass, nylon, carbon, etc. However it is also established that mechanical properties of natural fiber composites are much lower than those of synthetic fibers composite. Another disadvantage of natural fiber composite which makes them less attractive is the poor resistance to moisture absorption [77]. Hence use of natural fiber alone in polymer matrix is inadequate in satisfactorily tackling all the technical needs of a fiber reinforced composite. Hence in the present work an effort has been made to develop superior, but economical composite by combining natural fibers (Jute and Silk) with a synthetic fiber (E-Glass) in the same matrix material so as to take the best advantage of the properties of both the fibers. This results in a hybrid composite and the erosive wear behavior of the same has been carried out and the results are presented in the following sections.

5.3 MATERIALS AND METHOD

5.3.1 Raw Material Used

1. Silk Fiber
2. E-Glass Fiber
3. Jute Fiber
4. Epoxy Resin
5. Hardener

5.3.1.1 Silk Waste fiber

The silk fiber used in the present investigations details are explained in chapter 3 art 3.1.1.

5.3.1.2 Jute fiber

As explained earlier natural vegetable fibers have attracted worldwide attention as a potential reinforcement for composite because of their easy availability as a renewable resource, easy process

ability, low density, light weight, non abrasive, low cost and above all for their biofriendly characteristics. The jute fiber shown in Figure-5.1 is an important bast fiber and comprises bundled ultimate cells, each containing spirally oriented microfibrills bound together. The main component of jute fiber is cellulose which leads to higher stiffness. Other components of jute fiber are hemi-cellulose, lignin, pectin, waxy and water soluble substances.

Figure-5.1 Woven Jute fiber

5.3.1.3 E glass fiber

Glass is the most common fiber used in polymer matrix composites. Its advantage includes its high strength, low cost, high chemical resistance, and good insulating properties. In the present investigation E- glass fiber 360 roving supplied by saint Gobian ltd was used. The fibers wear cut to sizes 150x60 mm from the long sheet shown in Figure-5.2.

Figure-5.2 E-Glass fiber

5.3.1.4 Epoxy Resin and Hardener

It has been discussed in detail in chapter 3 art 3.1.2 and art 3.1.3.

5.4 PREPARATION OF COMPOSITES:

Hybrid laminates of different stacking sequence were prepared by hand lay-up technique. A wooden mold of 150x60x5 mm was used for manufacturing the composite. For quick and easy removal of the composite sheet a mold release sheet was put over the glass plate. Mold release spray

was also applied at the inner surface of the mold wall after it was set on the glass plate. Four groups of laminate composite samples with total 3 plies were manufactured by varying stacking sequence of silk, jute and glass fabrics as presented in table 5.1. Jute, silk and glass fabrics were pre-impregnated with the matrix material consisting of epoxy resin and hardener in the ratio of 10:1. Care was taken to avoid formation of air bubbles during pouring. Pressure was then applied from the top and the mold was allowed to cure at room temperature for 72 hrs. During the application of pressure some polymer squeezes out from the mould. For this, care has already been taken during pouring. After 72 hrs the samples were taken out of the mold, after curing the laminate was cut into required size of erosion and other mechanical tests by diamond cutter.

Table-5.1

Different stacking Sequence

S No	Stacking Sequence	Description
1	**GSG**	Three layers of laminates upper and lower layer consist of composite with E-glass reinforcement while the middle one consist of reinforcement of silk waste fiber.
2	**SGS**	Three layers of laminates upper and lower layer consist of composite with silk waste fiber reinforcement while the middle one consist of reinforcement of E-glass.
3	**JSJ**	Three layers of laminates upper and lower layer consist of composite with Jute mate reinforcement while the middle one consist of reinforcement of silk waste fiber.
4	**SJS**	Three layers of laminates upper and lower layer consist of composite with silk waste fiber reinforcement while the middle one consist of reinforcement of Jute mate.

5.5 TESTING OF MECHANICAL PROPERTIES.

The tensile strength, flexural strength, interlaminar shear strength, density and the micro hardness of the hybrid composite for different stacking sequences were found out as per the procedure explained in chapter-3 art-3.5. The characterization of hybrid composite is similar to the method adopted for different weight fraction of reinforcement that has been already described in details in chapter 3 and chapter art 3.4.1, art 3.4.2, art 3.4.6 and presented in Table-5.2 to Table 5.4.

5.6 SOLID PARTICLE EROSION TEST FOR HYBRID COMPOSITE.

The solid particle erosion test for different sequences for the hybrid composite were carried out as per ASTM G76 standard The erosion test rig and the experimental procedure ,test parameters, and the wear rate measurements wear same as explained in chapter 4 art 4.3 .The experimental results are presented in Table- 5.5 to 5.9.

Table-5.2

Density and micro hardness of different stacking sequence

Sample	Micro-hardness	Density (g/cm^3)
GSG	25.375	1.168
SGS	22.894	1.167
JSJ	20.315	1.182
SJS	22.114	1.178

Table-5.3

Tensile Strength

Stacking Sequence	Tensile Strength (MPa)	Tensile Modulus (GPa)
GSG	102.35	6.02
SGS	90.88	5.35
JSJ	79.17	4.54
SJS	87.51	5.15

Table-5.4

Flexural Strength, Flexural Modulus, ILSS

Stacking Sequence	Flexural Strength (MPa)	Flexural Modulus (GPa)	Interlaminar Shear Strength (MPa)
GSG	252.5	13.6	5.63
SGS	209.8	9.8	4.91
JSJ	217.3	11.3	4.97
SJS	201.7	9.2	4.86

Table-5.5

Erosion rate of all the samples at 30^0

Velocity (m/s)	Cumulative Weight (g)	Erosion Rate x 10^{-4} (g/g)			
		GSG	SGS	SJS	JSJ
48	40.5	0.568	1.23	1.52	1.98
	81	0.657	1.23	1.6	1.85
	121.5	0.823	1.32	1.56	1.65
	162	0.617	1.3	1.67	1.79
	202.5	0.987	1.19	1.58	1.98
70	40.5	2.47	2.52	2.7	3.1
	81	1.23	1.98	1.98	2.22
	121.5	1.65	1.74	2.14	2.22
	162	1.33	1.48	2.4	2.1
	202.5	1.48	1.33	2.27	2.6
82	40.5	2.47	3.14	2.9	4.94
	81	3.7	3.9	3.8	4.94
	121.5	3.29	4.12	3.29	5.76
	162	3.09	4.32	3.7	5.56
	202.5	2.96	3.95	4.5	5.43

Table-5.6

Erosion rate of all the samples at 45^0

Velocity (m/s)	Cumulative Weight (g)	Erosion Rate x 10^{-4} (g/g)			
		GSG	SGS	SJS	JSJ
48	40.5	1.98	2.11	2.33	2.88
	81	2.12	2.56	2.6	2.99
	121.5	2.44	2.66	2.98	3.29
	162	3.01	2.78	3.09	3.09
	202.5	2.96	2.45	3.29	3.46
70	40.5	2.41	2.47	2.94	4.94
	81	2.94	2.47	3.7	4.94
	121.5	3.19	3.29	4.12	5.76
	162	3.29	4.32	4.32	5.94
	202.5	3.09	3.95	3.95	5.43
82	40.5	2.47	4.94	4.94	7.41
	81	4.64	6.17	6.17	8.64
	121.5	5.73	5.76	6.58	10.7
	162	6.64	5.56	6.79	9.88
	202.5	6.81	5.93	6.91	9.38

Table-5.7

Erosion rate of all the samples at 60^0

Velocity (m/s)	Cumulative Weight (g)	Erosion Rate x 10^{-4} (g/g)			
		GSG	SGS	SJS	JSJ
48	40.5	0.584	2.22	1.73	2.47
	81.00	0.8	1.73	1.6	2.47
	121.5	1.23	1.56	1.65	3.29
	162.00	1.23	1.98	1.6	2.47
	202.50	1.48	2.12	1.73	2.96
70	40.5	1.23	2.47	1.41	4.12
	81	1.23	2.47	2.17	4.32
	121.5	1.89	3.29	2.76	4.44
	162	1.8	3.7	2.56	4.94
	202.5	2.9	3.46	2.43	4.94
82	40.5	3.94	7.41	4.94	7.41
	81.00	6.11	6.17	4.94	7.41
	121.5	5.05	5.76	5.76	9.88
	162.00	4.64	6.17	5.56	9.26
	202.50	4.4	6.42	5.43	9.38

Table-5.8

Erosion rate of all the samples at 90^0

Velocity (m/s)	Cumulative Weight (g)	Erosion Rate x 10^{-4} (g/g)			
		GSG	SGS	SJS	JSJ
48	40.5	1.47	1.98	1.73	2.47
	81.00	1.23	1.85	1.36	3.7
	121.5	1.65	1.65	1.4	3.29
	162.00	1.85	1.91	1.42	3.7
	202.50	1.98	2.07	1.33	3.46
70	40.5	1.47	4.94	2.47	4.94
	81.00	2.7	4.94	2.47	4.94
	121.5	3.12	4.12	2.47	4.12
	162.00	2.7	3.7	3.09	4.32
	202.50	2.46	3.46	2.96	4.94
82	40.5	2.88	2.47	4.94	5.12
	81	2.88	3.7	5.12	5.3
	121.5	3.05	4.13	5.23	5.35
	162	3.02	5.13	5.44	5.43
	202.5	3.4	5.56	5.66	5.56

Table-5.9

Erosion Efficiency

Impingement Angle	Velocity (m/s)	Erosion Efficiency (%)			
		GSG	SGS	JSJ	SJS
30^0	48	0.915	1.45	1.014	1.145
	70	1.429	2.135	1.141	1.963
	82	2.005	2.167	1.67975	2.1895
45^0	48	1.892	2.548	3.361	2.63
	70	1.925	2.642	4.829	3.179
	82	2.717	4.206	5.058	3.391
60^0	48	0.791	2.465	2.067	1.795
	70	1.617	4.652	2.302	5.379
	82	1.776	4.994	2.353	5.445
90^0	48	1.361	2.522	1.721	3.095
	70	1.557	3.725	1.842	3.56
	82	1.82	5.661	1.937	5.857

Table-5.10

Parameters characterizing the velocity dependence of erosion rate of Epoxy and its composites.

Samples	Impingment Angle(α°)	k	n	R^2
GSG	30	$9x10^{-7}$	3.4122	0.9959
	45	$4x10^{-5}$	2.7448	0.9892
	60	$8x10^{-8}$	4.224	0.9772
	90	$5x10^{-5}$	2.6906	0.8779
SGS	30	$1x10^{-3}$	1.8353	0.837
	45	$5.4x10^{-3}$	1.5647	0.8129
	60	$2.1x10^{-3}$	1.8212	0.6226
	90	$3.5x10^{-3}$	0.5632	0.769
JSJ	30	$2.2x10^{-3}$	1.713	0.7053
	45	$1.2x10^{-3}$	2.0072	0.946
	60	$3x10^{-4}$	2.25	0.9998
	90	$1.1x10^{-3}$	1.9215	0.9893
SJS	30	$1.8x10^{-2}$	1.166	0.858
	45	$1.9x10^{-2}$	1.2803	0.98
	60	$1.4x10^{-3}$	1.7977	0.5592
	90	$3x10^{-4}$	2.1735	0.9499

5.7 RESULT AND DISCUSSION

5.7.1 MECHANICAL PROPERTIES

5.7.1.1 Micro Hardness

The present investigation reveals that by varying the number and position of glass and silk layers and jute and silk layer four different stacking sequences are obtained as shown in Figure-5.3. The composite micro-hardness is different for different stacking sequences. Its maximum value is for sequence GSG.

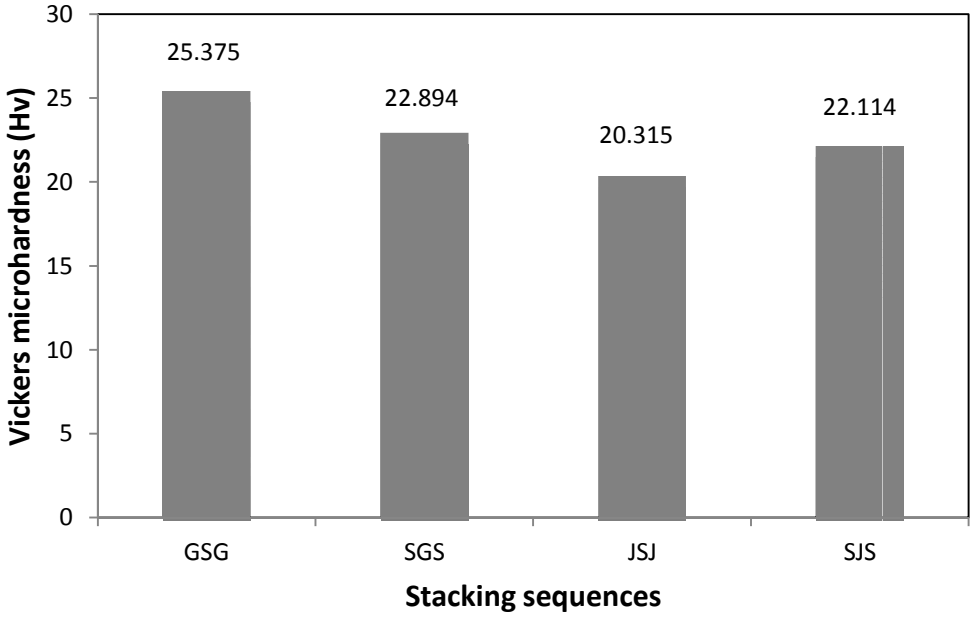


Figure-5.3 Effect of hardness on stacking sequence in epoxy composite

5.7.1.2 Tensile Strength and Modulus

The tensile strength and modulus of unreinforced epoxy resin is found to be 18.03MPa and 521Mpa. The variation of tensile strength and modulus for various laminate stacking sequences is shown in Figure-5.4 to 5.5. The tensile strength and modulus of the composite is influences by the strength and modulus of fiber [78]. In our case the tensile strength and modulus of laminate with GSG samples shows the highest value. The increase in the tensile strength and modulus of GSG composite is attributed to the fact that, glass fibers are stronger and stiffer than silk fibers. An increase in the

tensile strength and modulus of 23% and 4% is observed for GSG and SGS sample compared to JSJ and SJS samples.

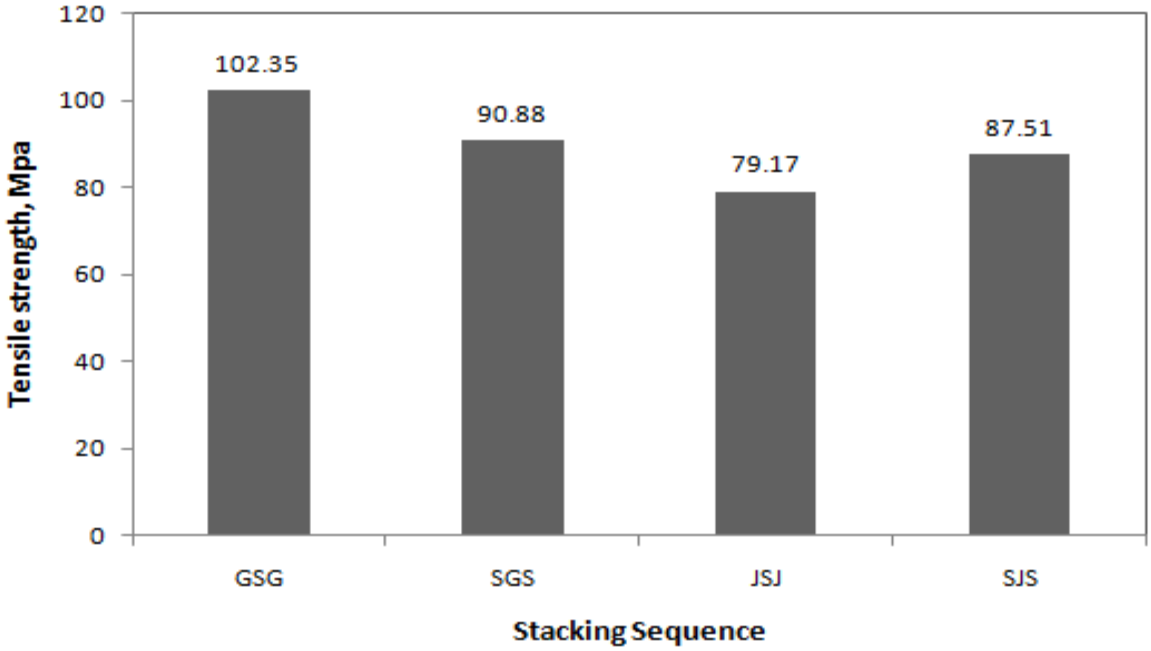


Figure-5.4 Variation of tensile strength for stacking sequence in epoxy composite

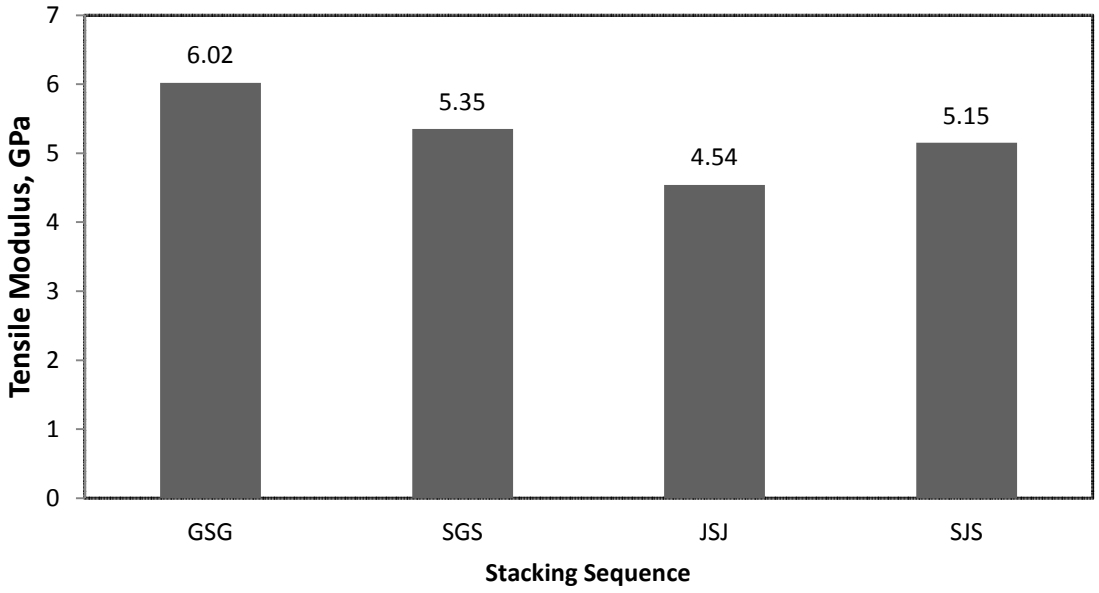


Figure-5.5 Variation of tensile modulus for stacking sequence in epoxy composite

5.7.1.3 Flexural Strength and Inter laminar shear stress.

The Flexural strength, modulus and Inter laminar shear stress for laminates with different stacking sequences are shown in Figure-5.6 to 5.8 respectively. It has been observed that GSG sample has highest value of flexural stress, flexural modulus as well as for Inter laminar shear stress value which is found to be 252.5MPa, 13.6GPa and 5.63 MPa respectively. Which is 20% higher than the SGS samples which has the lowest value found for all these strengths (201.7 MPa, 9.2 GPa and 4.86 MPa respectively).

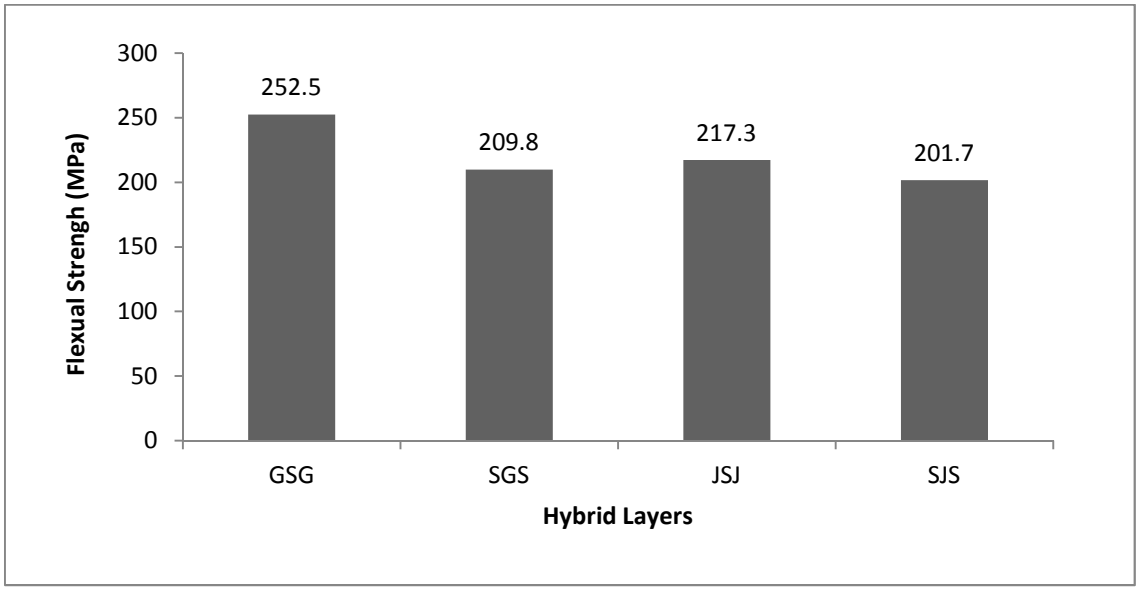


Figure-5.6 Flexural strength of Different samples

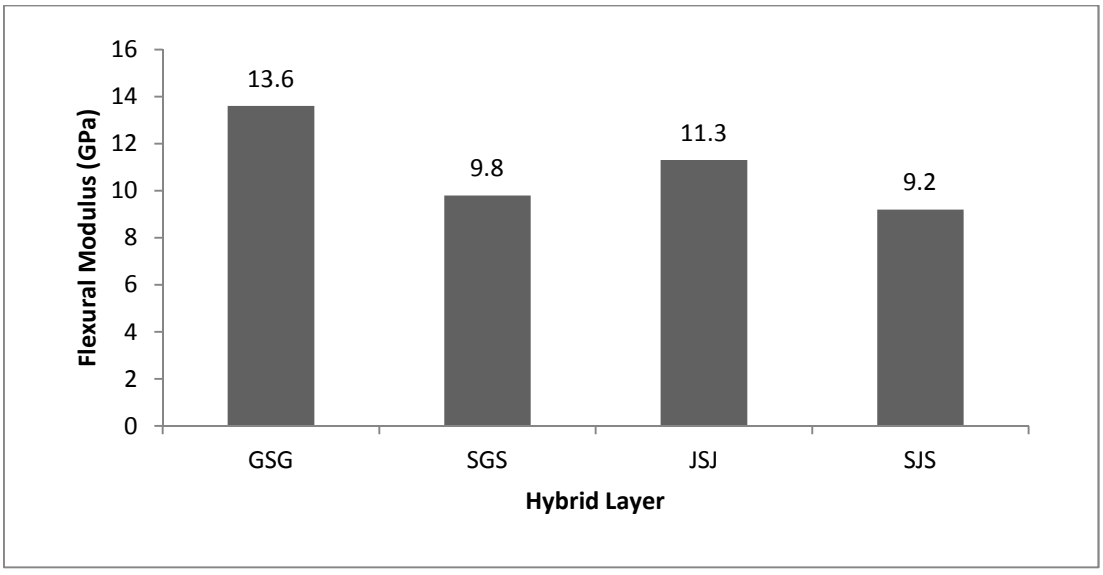


Figure-5.7 Flexural Modulus of Different samples

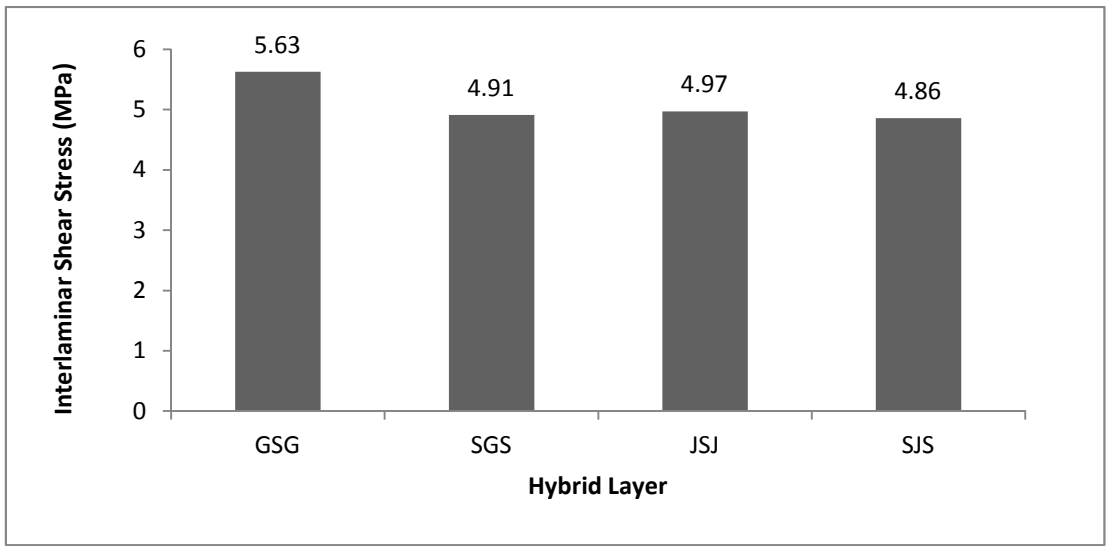


Figure-5.8 ILSS of Different samples

5.7.1.4 Density

The densities of different layered samples are shown in Figure-5.9. The layered SGS sample has shown the lowest density among all having its value 1.167gm/cm^3.

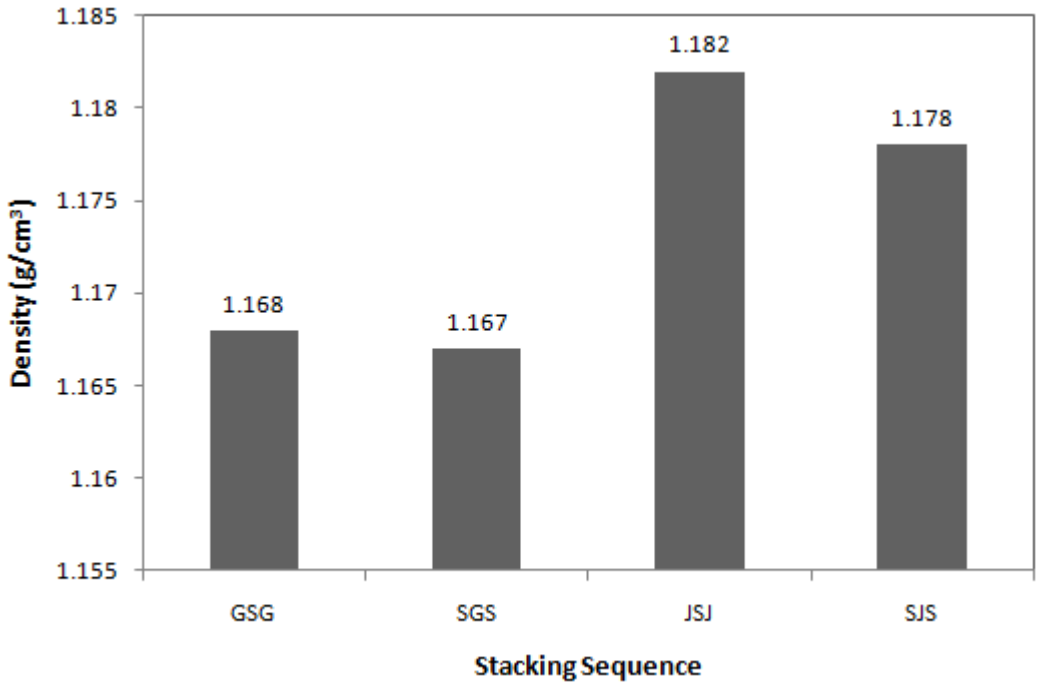


Figure-5.9 Density of various laminate comosites

5.7.2 EROSION RATE:

Figure-5.10 to 5.21 shows variation of erosion rate of all the sequential layered composites as a function of the cumulative weight of impinging particle with different impinging angles (30^0,45^0,60^0 & 90^0) and with different impact velocities (48, 70, 82, m/s). The plots were obtained by determining the steady state of the weight loss. The cumulative weight of impinging particle to achieve steady state value was varying with material. The nature of the curve is also different for different materials tested. In all these curves no incubation or induction period is observed for different material tested. Instead these figures clearly show that the response of material to the weight of the erodent was acceleration, peaking, deceleration and stabilization.

In order to study the effect of particle velocity on erosion rate, erosion tests were performed by varying the particle velocity 48, 70 and 82m/s for various impingement angles (30^0–90^0). Figure-5.22 to 5.25 shows the typical steady-state erosion rate dependence of epoxy and its composites for different impact velocities at four impingement angles (30^0,45^0,60^0 and 90^0). It can be observed from these figures, that erosion rate of all laminates increases with increase in impact velocity for different impingement angles. If the solid particle impact experiments the impact velocity of the erosive particles has a very strong effect on erosion rate. For any materials, once the steady state conditions have been reached the erosion rate 'E_r' can be expressed as a simple power function of impact velocity(v)[74]

$$E_r = kv^n \qquad (5.1)$$

where *k* is the constant of proportionality includes the effect of all the other variables. The value of '*n*' and '*k*' are found by least-square fitting of the data points in plots which represent the erosion rate dependence on impact velocity by using the power law. The value of *'n'*, the velocity exponent, is typically between 2 and 3, although much higher exponent is seen under some circumstances [56]. According to Pool et al. [74], for polymeric materials behaving in ductile manner, the velocity exponent *'n'* varies in the range 2-3 while for polymer composites behaving in brittle fashion the value of *'n'* should be in the range of 3-5. Figure 5.26 to 5.29 illustrates the variation erosion rate with impact velocity at different impingement angle for neat epoxy and its composites. The least-square fits to data point were obtained by using power law and the values of '*n*' and '*k*' are summarized in Table-5.10. The velocity exponents found for 30^0, 45^0, 60^0 and 90^0 impingement angles

are in the range of 1.166–3.4122, 1.2803-2.7448, 1.7977-4.224 and 0.5632–2.6906 respectively. This velocity exponent at various impingement angles are in conformity with Harsha et al. [51.].

Figure-5.30-5.32 shows the influence of impingement angle (α) on the erosion rate of hybrid composite. This shows that peak erosion takes place at impingement angle of 45^0 for all laminates. As discussed earlier in the chapter4 art 4.3, for the hybrid composite since the maximum erosion rate is at 45^0 impingement angle it can be said that these composites are neither behaving in a purely ductile nor in a purely brittle manner. So this behavior of these composites can be termed as semi-ductile in nature.

5.8 SURFACE MORFOLOGY OF ERODED SAMPLES

Figure-5.33 shows the eroded surface of GSG sample. Figure-5.33 (a) shows the crater formed and the damaged caused to the fiber. It shows extensive damage of the fiber. Fibers bending are visible instead pulling out of fiber from the matrix. Figure- 5.33(b) shows the broken and semi-broken fiber within the composites. Though the fibers are broken, are not chipped off from the matrix This may be due to good binding between fiber and matrix, also the presence of silk fiber at the middle to some extent prevents the pulling out of glass fiber from the matrix.

5.9 CONCLUSION

The following conclusions are drawn from the above studies.

1. Hybridization increases strength of the silk fiber epoxy composite.
2. Incorporation of glass in silk fiber composites enhances the properties of resulting hybrid composites in comparison to jute fiber inclusion with silk fiber.
3. Layering sequence (altering the position of jute/silk/glass sequence) significantly affects the flexural and inter-laminar shear strength and erosive properties.
4. For the same relative weight fraction of silk and glass (or silk and jute) fiber, layering sequence has little effect on tensile, flexural and erosive properties.
5. Overall comparison between the properties of all the laminates revealed that the hybrid laminate with two extreme glass plies on either side (GSG) is the optimum combination with a good balance between the properties and cost.

6. The velocity of the erosive particles has a very strong effect on erosion rate. It was found that the erosion rate follows power law behavior with particle velocity, $R \alpha V^n$ and the velocity exponent 'n' was found in the range of 1.173–3.4122.
7. The influence of impingement angle on erosive were of all composites under consideration exhibit semi ductile behavior with maximum erosive wear rate at 45^0 impingement angle.
8. The erosion efficiencies vary from 0.791% to 5.445% for different impact velocities and angles.
9. It is clear from this study that erosive strength of natural fiber (silk) can be increased by hybridization with synthetic fiber.

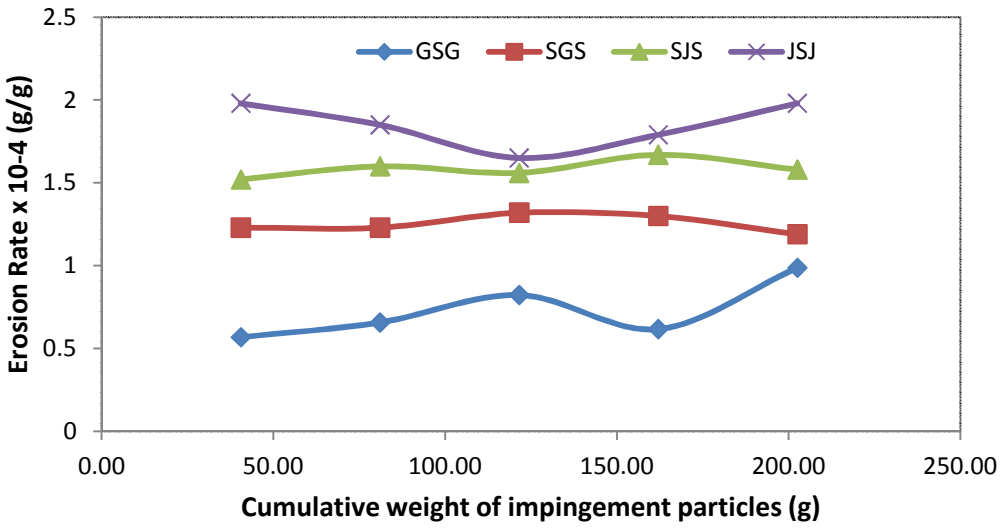


Figure-5.10 Variation of erosion rate with cumulative weight of impingement particle at impingement angle 30^0 at velocity 48m/s.

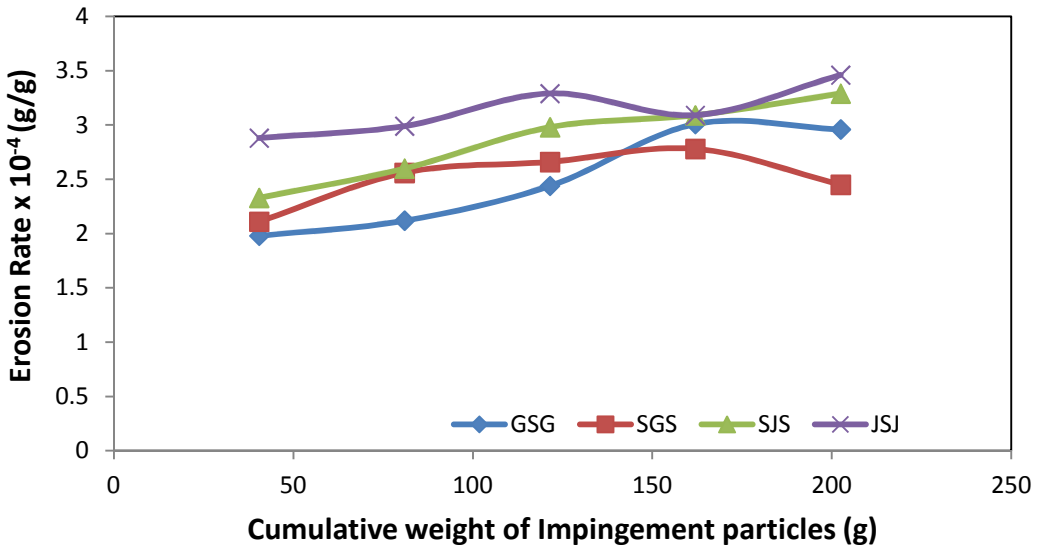


Figure-5.11 Variation of erosion rate with cumulative weight of impingement particle at impingement angle 45^0 at velocity 48m/s.

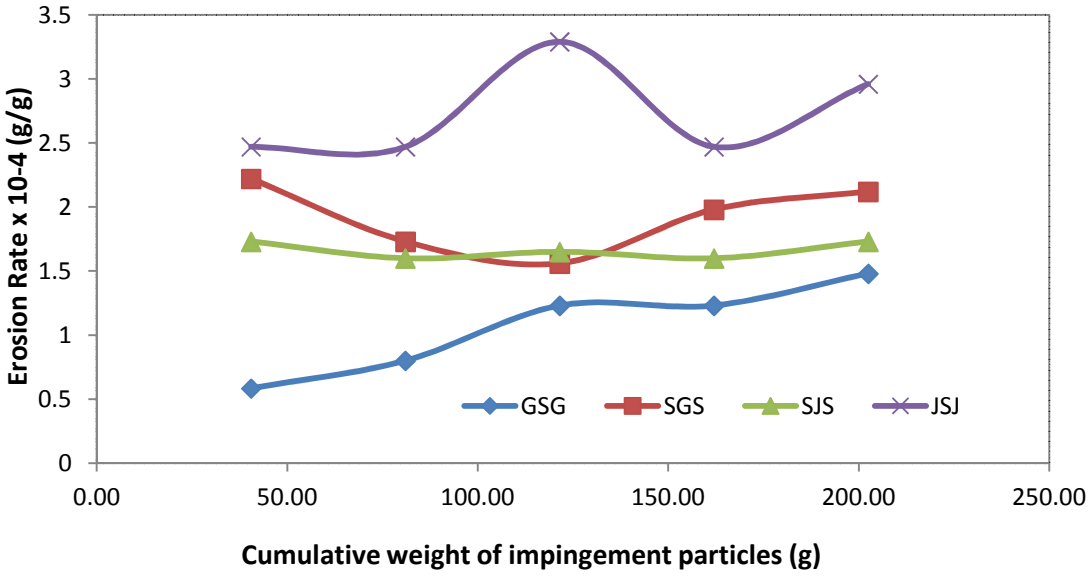


Figure-5.12 Variation of erosion rate with cumulative weight of impingement particle at impingement angle 60^0 at velocity 48m/s.

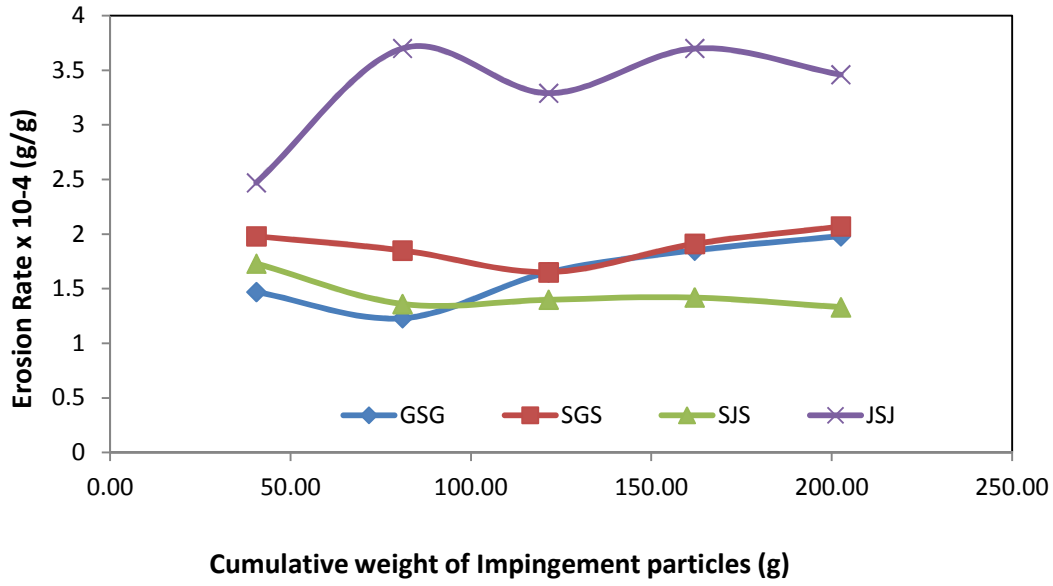


Figure-5.13 Variation of erosion rate with cumulative weight of impingement particle at impingement angle 90^0 at velocity 48m/s.

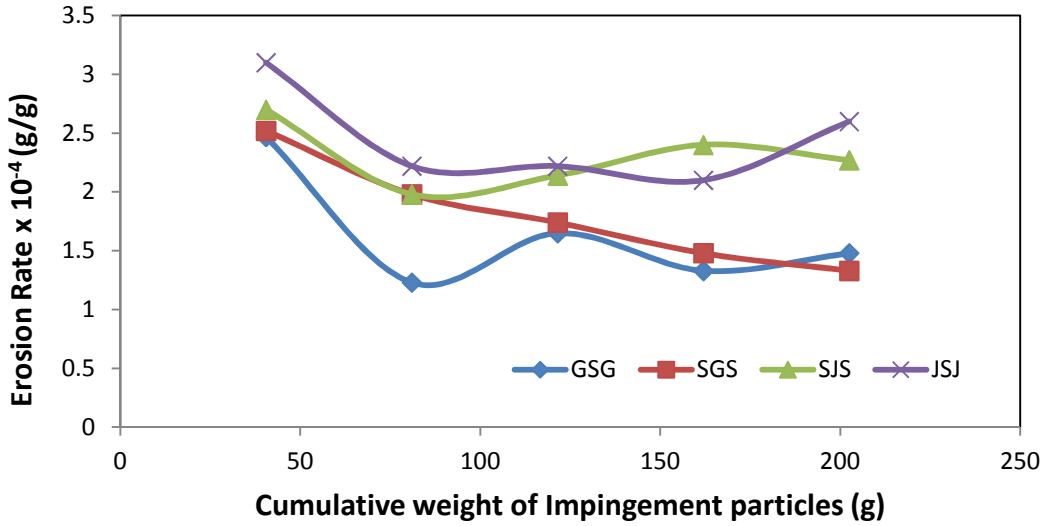


Figure-5.14 Variation of erosion rate with cumulative weight of impingement particle at impingement angle 30^0 at velocity 70m/s.

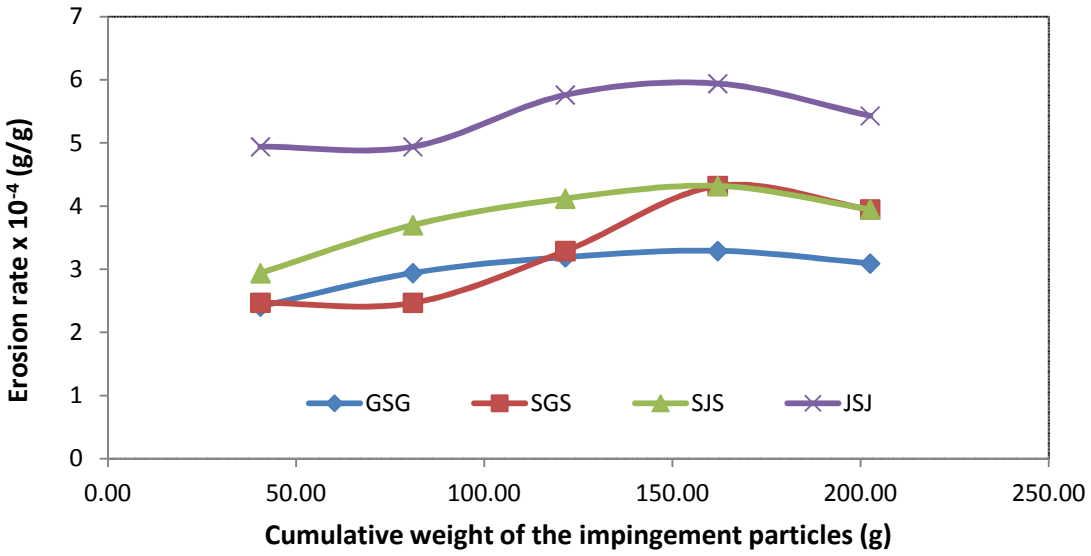


Figure-5.15 Variation of erosion rate with cumulative weight of impingement particle at impingement angle 45^0 at velocity 70m/s

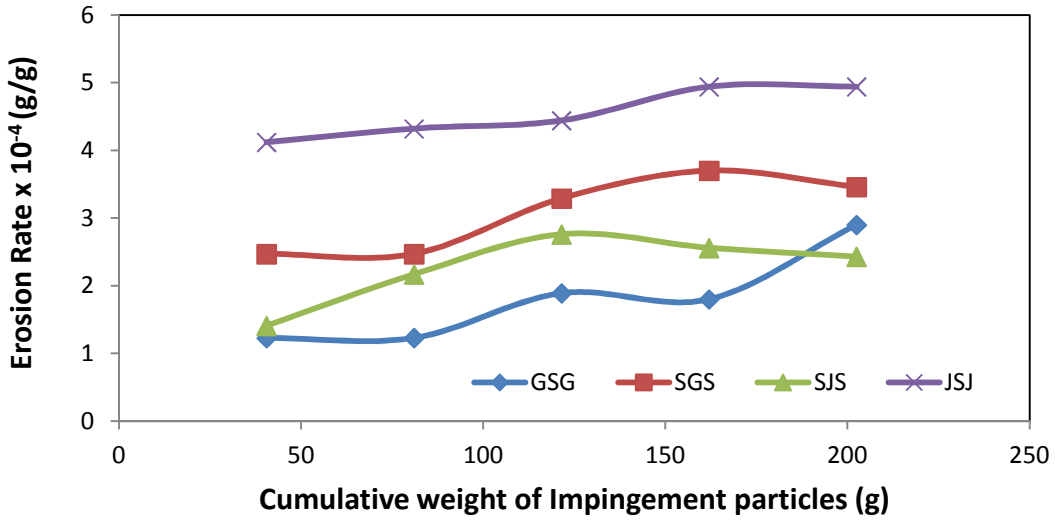


Figure-5.16 Variation of erosion rate with cumulative weight of impingement particle at impingement angle 60^0 at velocity 70m/s

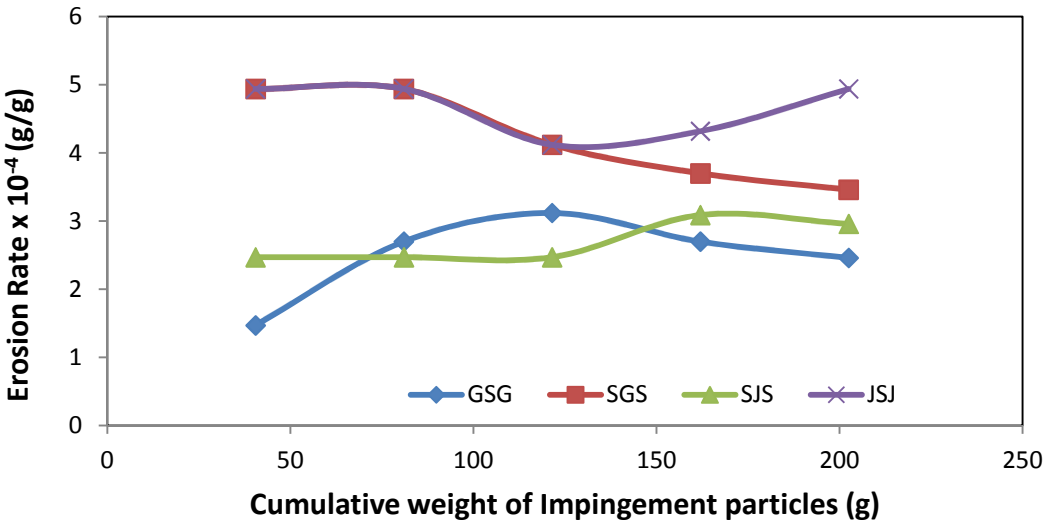


Figure-5.17 Variation of erosion rate with cumulative weight of impingement particle at impingement angle 90^0 at velocity 70m/s

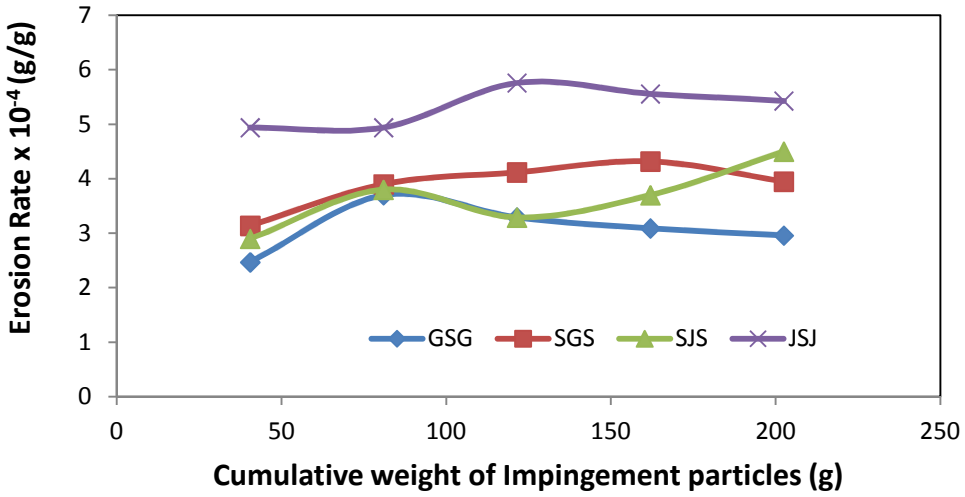


Figure-5.18 Variation of erosion rate with cumulative weight of impingement particle at impingement angle 30^0 at velocity 82m/s

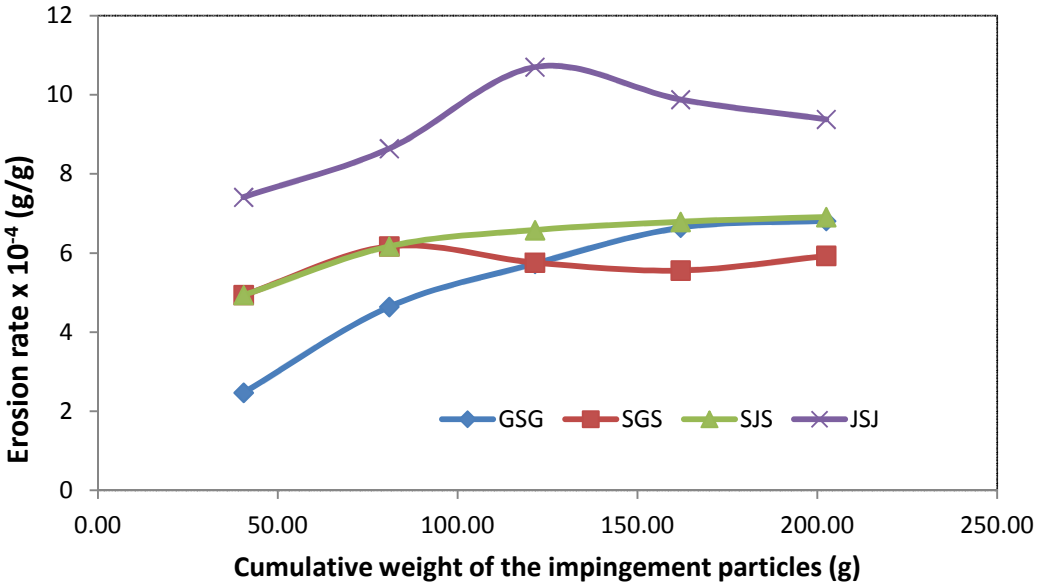


Figure-5.19 Variation of erosion rate with cumulative weight of impingement particle at impingement angle 45^0 at velocity 82m/s.

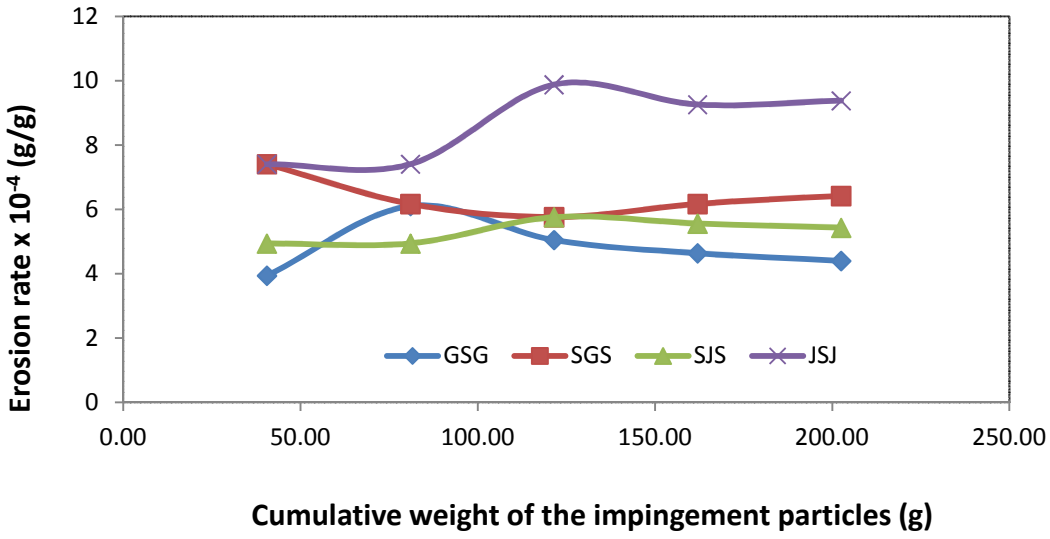


Figure-5.20 Variation of erosion rate with cumulative weight of impingement particle at impingement angle 60^0 at velocity 82m/s.

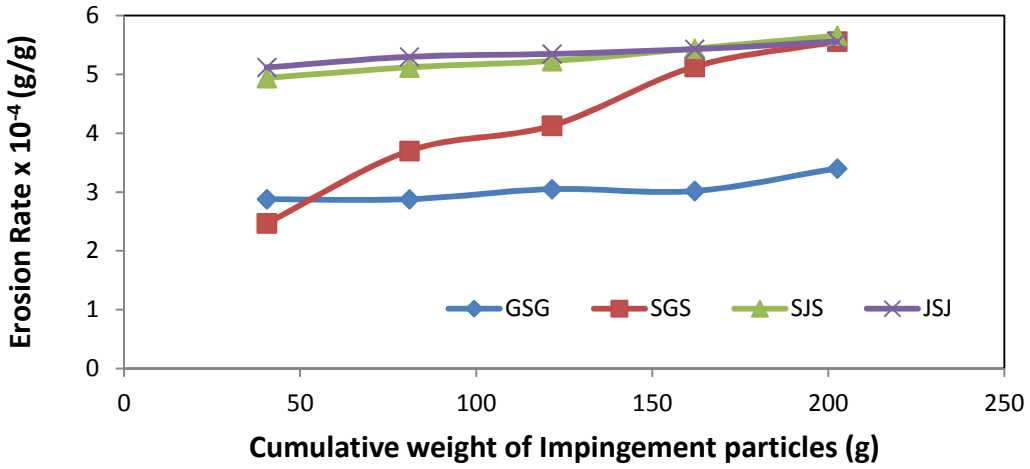


Figure-5.21 Variation of erosion rate with cumulative weight of impingement particle at impingement angle 90^0 at velocity 82m/s.

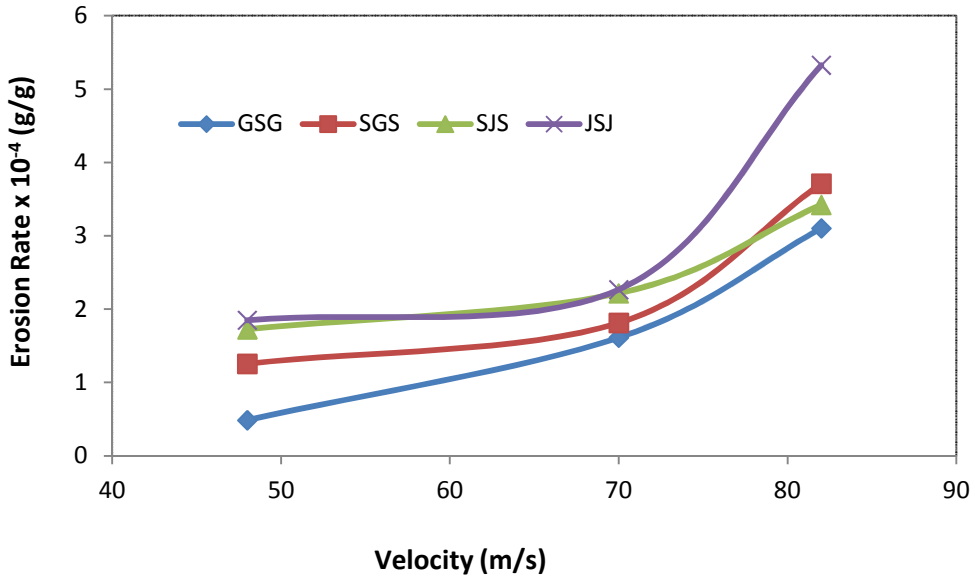


Figure-5.22 Variation of erosion rate with velocity of particle at impingement angle 30^0

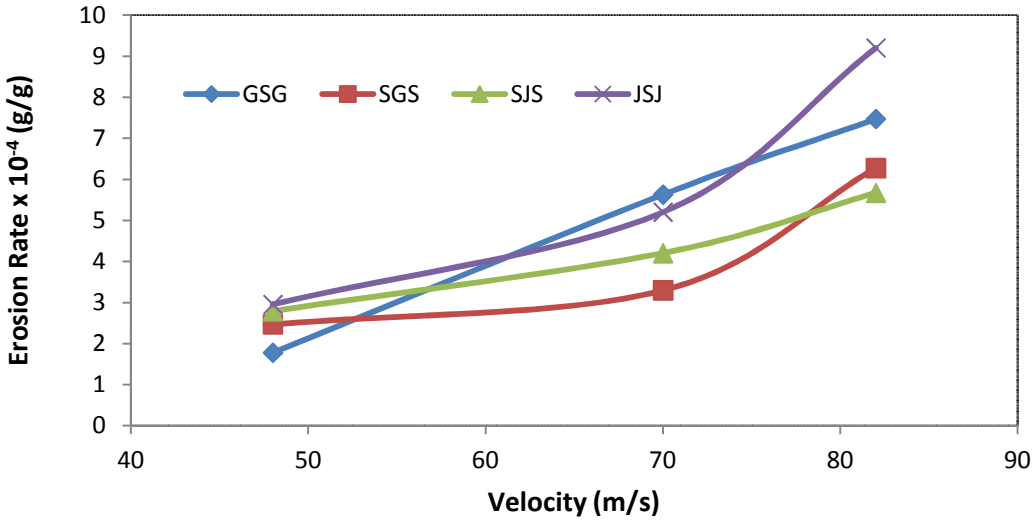


Figure-5.23 Variation of erosion rate with velocity of particle at impingement angle 45^0

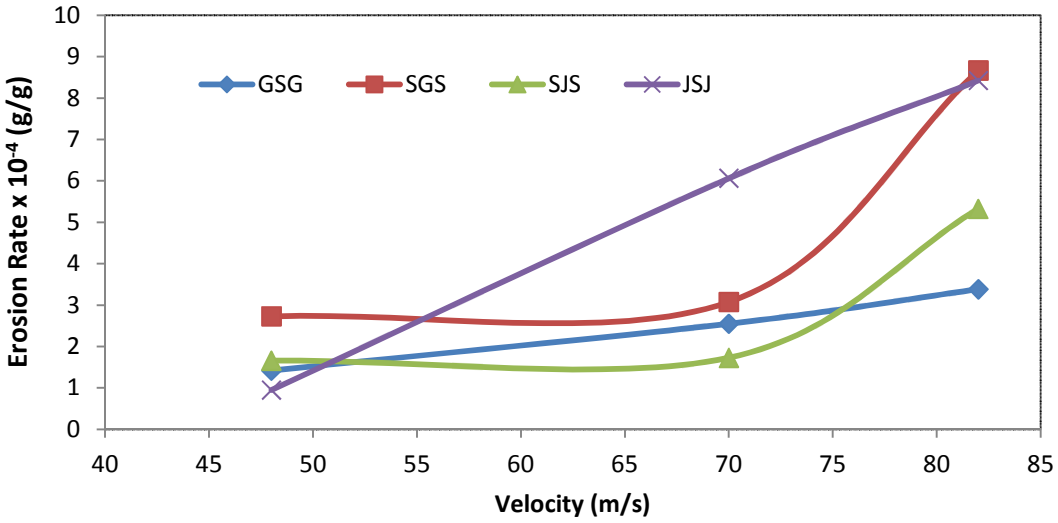


Figure-5.24 Variation of erosion rate with velocity of particle at impingement angle 60^0

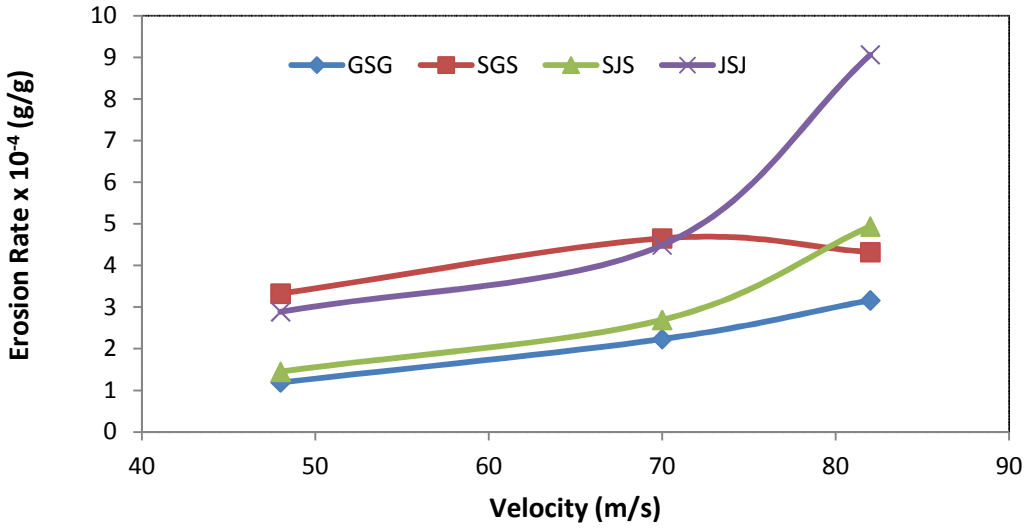


Figure-5.25 Variation of erosion rate with velocity of particle at impingement angle 90^0

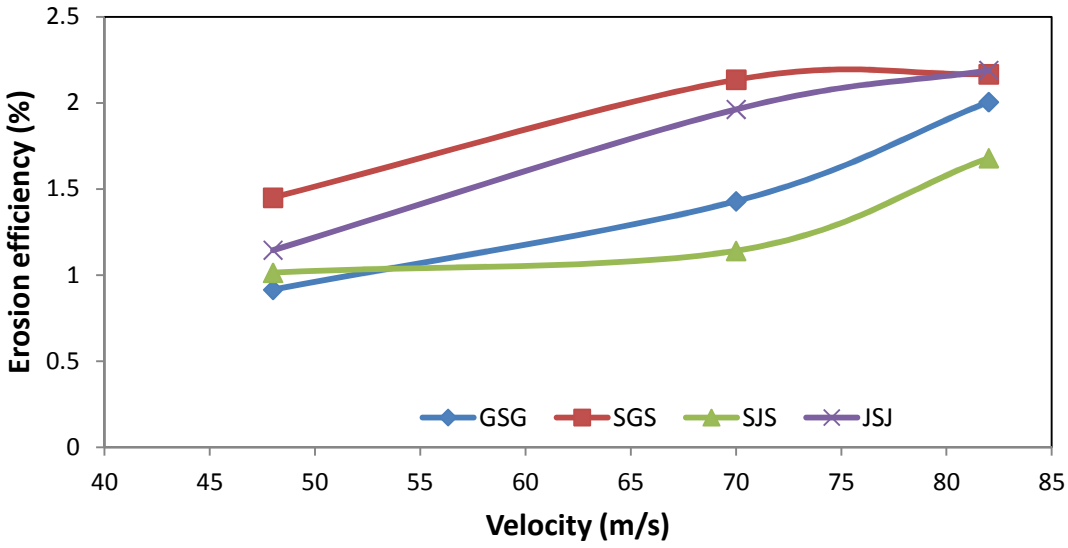


Figure-5.26 Variation of erosion efficiency with different velocity at impingement angle 30^0

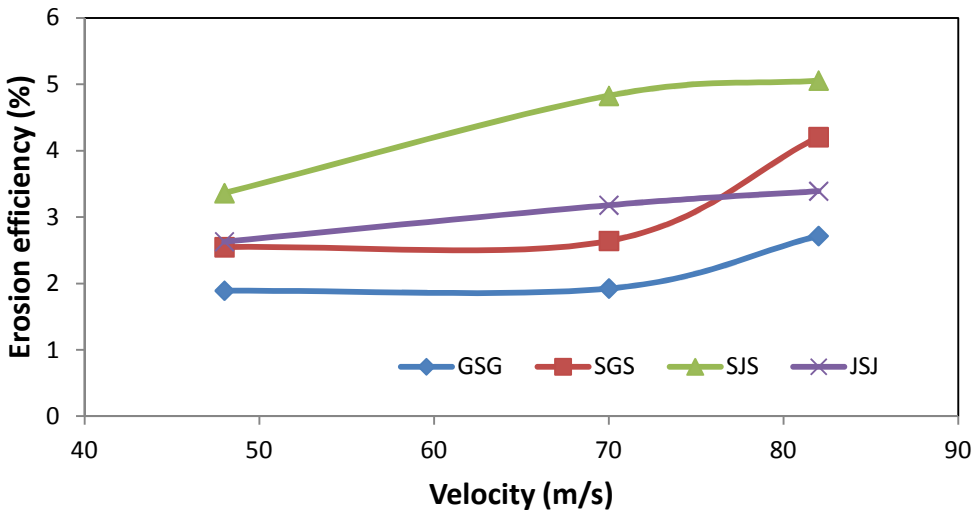


Figure-5.27 Variation of erosion efficiency with different velocity at impingement angle 45^0

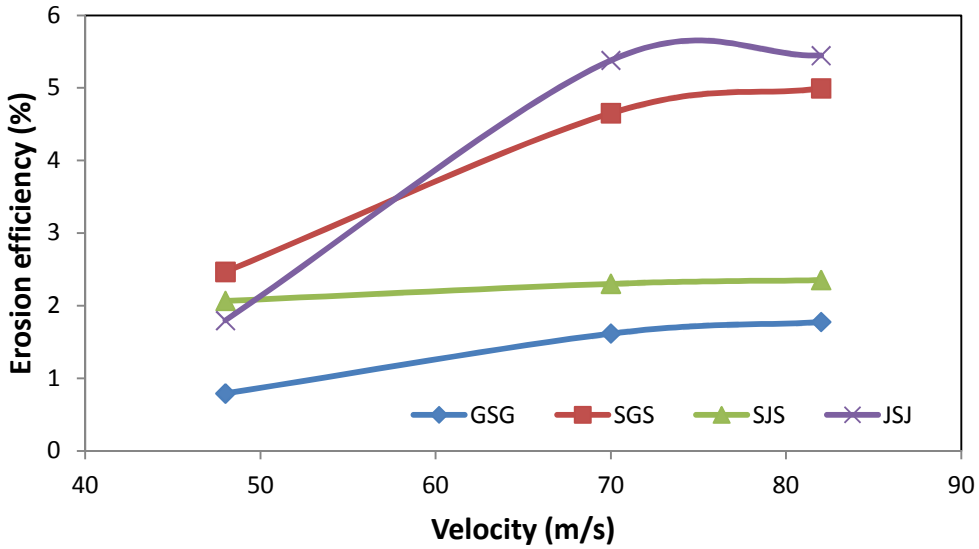


Figure-5.28 Variation of erosion efficiency with different velocity at impingement angle 60^0

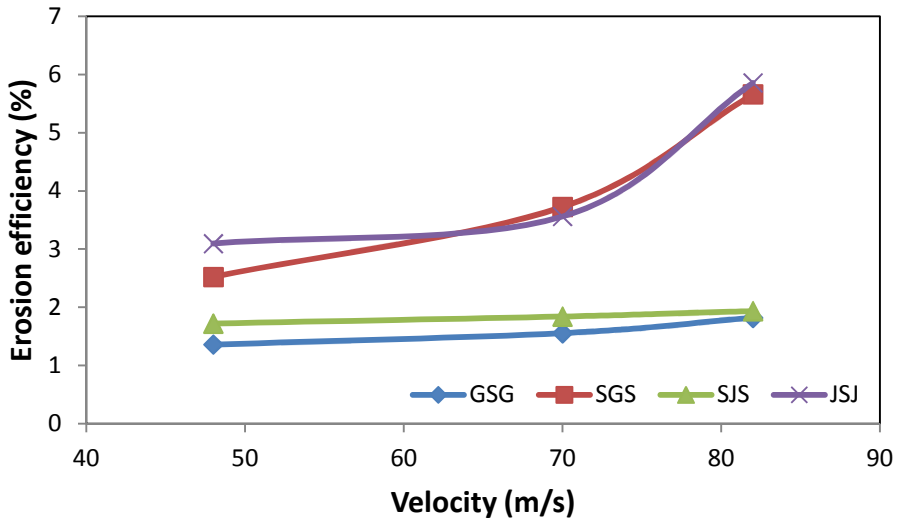


Figure-5.29 Variation of erosion efficiency with different velocity at impingement angle 90^0

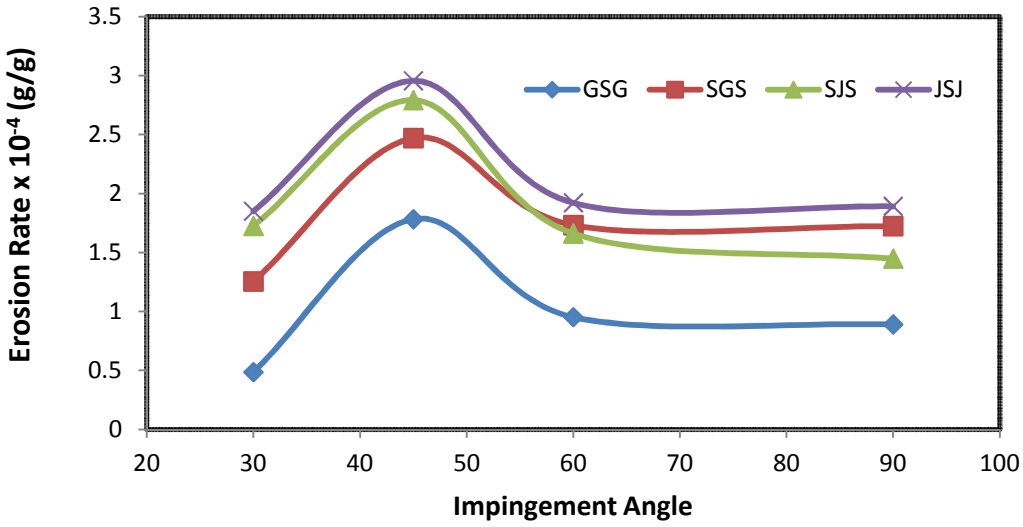


Figure-5.30 Variation of erosion rate with different impingement angle at velocity 48 m/s

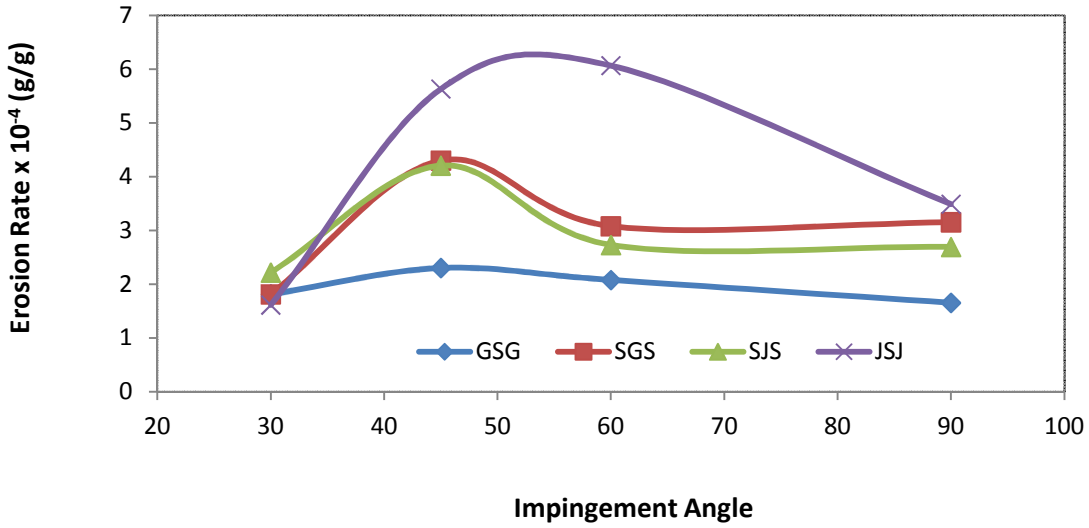


Figure-5.31 Variation of erosion rate with different impingement angle at velocity 70 m/s

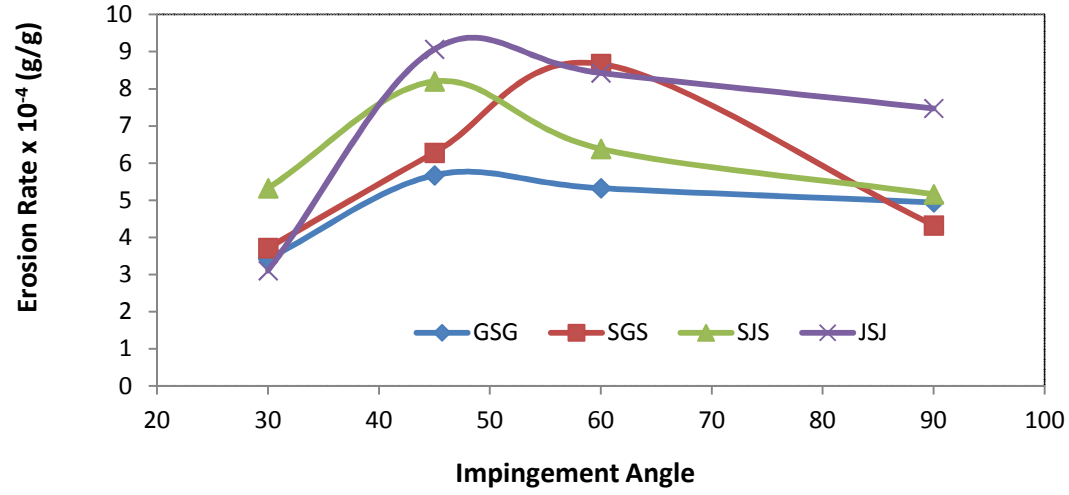
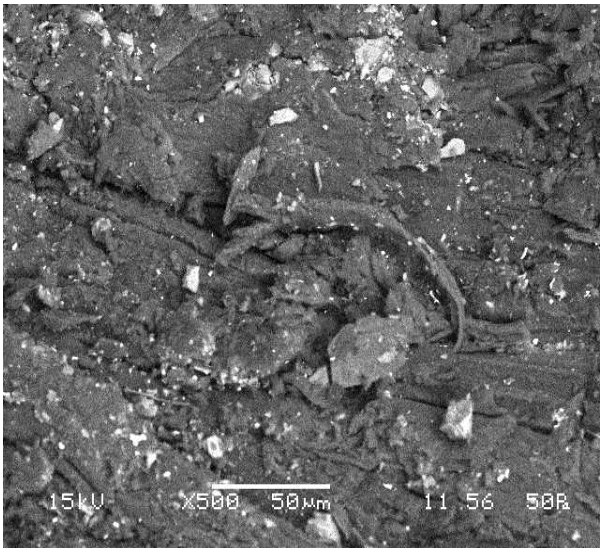


Figure-5.32 Variation of erosion rate with different impingement angle at velocity 82 m/s



(a)

Figure-5.33 (a) SEM micrograph of GSG sample at 45^0 impingement angle

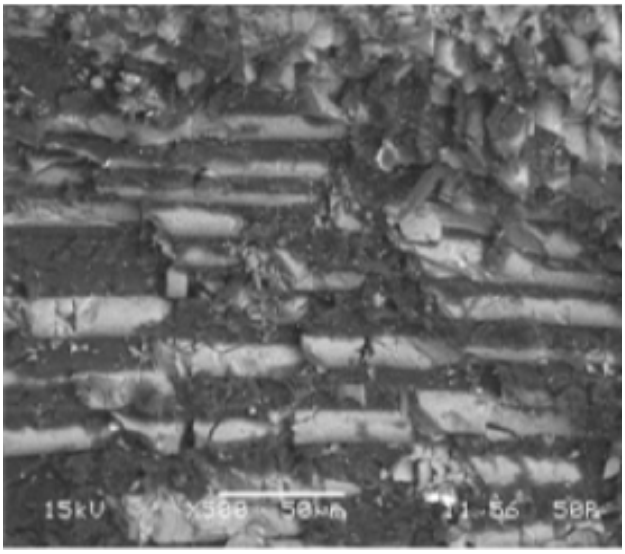


Figure-5.33 (b) SEM micrograph of GSG sample at 60^0 impingement angle

CHAPTER-6

CONCLUSIONS

6.1 CONCLUSIONS

The present work deals with the preparation of characterization of waste silk fiber reinforced epoxy composite. The experimental investigation in to weathering and erosion behavior of the composite lead to the following conclusions

1. With the successful fabrication of a new class of epoxy based composites reinforced with silk fiber and subjecting them to different environment it is found that the moisture absorption of the composite gets stabilized after certain period of exposure in all types of environment.

2. The flexural strength of the composite is found to be maximum with 4 volume percent of silk fiber for all treatments. When the type of treatments is considered it is found that the composite subjected to saline treatment shows higher strength. This might have occurred due to the formation of mono layers which formed due to electron rich species with sodium ions.

3. The erosive response of the silk fiber composites shows semi ductile behavior. From the experiment the erosion efficiency ($\boldsymbol{\eta}$) is found in the range 1.273% to 6.092%

4. Hybridization improves the erosive response of the silk fiber from semi ductile to ductile nature. The erosion efficiency for hybrid composite varies from 0.915% to 5.44%

5. It is ascertain from the present investigation that mechanical and erosive properties of natural fiber can significantly be improved by in cooperating synthetic fiber with it.

6. SEM observation reveals that most of the fibers were broken instead of pulling out from the matrix. This indicates a good bonding between fiber and the matrix.

6.2 RECOMMENDATION FOR FURTHER RESEARCH

- In this study fiber weight fraction of 8% has been used. This can be further increased to higher weight fraction of fiber using other manufacturing methods.
- The current study is limited to erosion study only. It can be extended to other tribological tests.
- In the current study different tribological tests has been carried out on the untreated silk fiber epoxy composite. The same work could be extended to treated fiber composite.
- In the erosion test sand particle of 200±50 microns only have been used. This work can be further extended to other particle size and types of particle like glass bead etc, to study the effect of particle size and type of particles on wear behavior of the composite.

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